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ASPHALT-AGGREGATE INTERACTIONS IN HOT RECYCLING

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This report summarizes results of an investigation of asphalt-aggregate interactions in hot recycled systems. Materials used in the research included a severe stripping aggregate and a nonstripping aggregate. Both were evaluated using the Lottman 70 percent retained tensile strength criteria. Additional materials included a 40/60 RAP new aggregate system, one virgin asphalt, two RAP recovered binders, one modifier or recycling agent, and two blends. The modifier was selected using a recently developed specification involving physical, composition, and solubility properties.

Aggregates were evaluated for surface area, bulk composition, water soluble ions, cation exchange capacity, gradation, and specific gravities. Binders were tested for physical properties and composition properties using a modified Clay-Gel procedure, and compatibility properties using a modified Heithaus procedure. Asphalt-aggregate mixtures were tested for dry tensile strength, dry resilient moduli, wet tensile (Continued)

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strength and wet resilient moduli. Water damage resistance of the mixtures was evaluated using the NCHRP 274 procedure. Mixtures were tested for bonding energy using microcalorimetry.

Results indicate that the severe stripping aggregate had low surface area, high water soluble ion concentration per square meter, and was highly acidic. The nonstripping aggregate had a higher surface area, low water-soluble ion concentration per square meter, and was basic in composition. The 40/60 aggregate had the highest surface area, lowest water-soluble ion concentration per square meter, and was basic in composition.

The mixture results showed that recycled systems resisted the action of water better than virgin systems. Bonding energy measurements indicated that asphalt-aggregate mixtures with recycled binders released higher energy levels than mixtures with virgin and RAP binders. Energy release was shown to relate to aggregate surface area, porosity, and composition.

A model two-way ANOVA analysis involving strength parameters showed that aggregate, binder, and aggregate-binder terms were all statistically significant. Results of the study were used to propose material selection guidelines for hot recycling applications.

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PREFACE

This technical report has been reviewed and is approved for publication.

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SECTION I
INTRODUCTION

A. OBJECTIVE

The objective of this research was to investigate asphalt-aggregate interactions in hot recycling by conducting fundamental studies and to use the results as a basis for developing guidelines to improve material selection for asphalt recycling applications.

B. BACKGROUND

Bonding between asphalts and aggregates in paving mixtures is inadequately understood because of the complexity and variability of asphalts and aggregate surfaces. Adhesion of asphalts to aggregates is a surface phenomenon controlled by the physical and chemical properties of the component materials. Since the early 1900s, investigations have attempted to study this surface interaction through such techniques as: (1) scraping and peeling (References 1 and 2), (2) contact angle (Reference 1), (3) water stripping (References 3 and 4), (4) inverse gas-liquid chromatography (Reference 5), (5) infrared spectroscopy (References 6 and 7), (6) the Gagle procedure (Reference 8), and (7) microcalorimetry (Reference 9). Observations indicate that viscosity or penetration effects of binders alone cannot account for pavement performance problems; yet, recycling is conducted by restoring aged pavements based soley on considerations of viscosity or penetration or both.

Few studies have been reported to investigate asphalt-aggregate interactions in hot recycling. There are no guidelines incorporating the beneficial aspects of proper binder rejuvenation into the design process. The complexity of the materials has been identified (Reference 10) as a probable cause for the absence of such guidelines; however, hot recycling has become common practice in highway and airfield reconstruction programs.

·Highway (and some Air Force) pavement rehabilitation efforts have reported financial savings of 25 to 40 percent from using recycled instead of virgin materials. Although many highway departments engage in recycling projects, problems identified by Blaschke (Reference 11) persist such as

difficulty in (1) characterizing the component materials in the pavement, (2) excluding moisture effects in the pavement, and (3) maintaining quality control throughout the project.

Through the study of asphalt-aggregate interactions in hot recycling, this research will attempt to gain a fundamental understanding of the properties of recycled mixes that affect performance. Performance in this research is measured by the retained tensile strength after water damage. The results obtained will be used to stress the need to develop test methods or test programs for characterizing the component materials in a pavement targeted for hot recycling.

This research investigates the following:

*   Bonding characteristics of Recycled Asphalt Pavement (RAP)
*   Effect of a recycling agent on the bond behavior
*   Moisture-resistance characteristics
*   The characteristics of both strength and stiffness of recycled mixtures.

The basis for comparison is a control mixture consisting of a virgin asphalt and a virgin or reference aggregate.

C. SCOPE

The scope of this effort consisted of a state-of-the-art technology review of asphalt-aggregate interactions and the methods used for their evaluation. This review led to the selection of test procedures for characterization of the interaction between asphalt and aggregate in a hot recycling environment. The methods used in this study were applied to a virgin asphalt, two recovered RAP binders, a modifier, two recycled blends, and three aggregates. The results were analyzed and correlations identified between performance and material properties. This report documents the findings, conclusions, and recommendations, for continued research in this area. Guidelines for the selection of materials for hot recycling projects are proposed.

D. CONDUCT OF RESEARCH

This research consisted of two phases. Phase I consisted of a literature review, preliminary materials selection and evaluation, parameter definition and test matrix determination. Phase II consisted of identification and acquisition of additional materials, development of testing methods, final test matrix configuration, evaluation of the test matrix, analysis of results, development of guidelines, conclusions and recommendations.

In Phase I, a literature review was conducted to identify techniques applicable for studying asphalt-aggregate interactions in hot recycling. Materials were collected from three air force bases (MacDill, Nellis, and Tyndall) and Hurlburt Field where hot recycling projects were under consideration, ongoing, or recently completed. These materials were tested and the results from these tests provided the basis for the work conducted in Phase II.

In Phase II, the test matrix was constructed to include five binders, three aggregates, and one modifier. The binders were a virgin AC-30, two RAP binders, and two recycled blends. The blends were prepared, using the two RAP binders and one modifier, which was selected using the criteria established in ESL-TR-84-47 (Reference 12). Two aggregates from Georgia were chosed, based on their past performance record as documented by the Federal Highway Administration (FHWA) and the Georgia Department of Transportation (DOT). Previous experience with these materials in other studies (Reference 13) also aided in their selection. The third aggregate was a combination of 40 percent RAP aggregate to 60 percent new aggregate. This aggregate blend was chosen to mimic the actual recycling formula used at Nellis AFB although no prior performance data for this aggregate were avaliable. Aggregates were tested for chemical, physical, and surface properties. Asphalts were tested for physical, chemical, and solution properties. Bonding energies between binders and aggregates were measured and mixtures were tested for water resistance. Resilient modulus and tensile strength testing before and after water treatment was conducted to provide a measure for performance of the mixtures. Correlations between material and mixture properties were identified and used to propose guidelines for selection of materials for hot recycling projects.

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SECTION II
LITERATURE REVIEW

A. INTRODUCTION

The performance characteristics of asphalt-aggregate paving mixtures
are largely determined by the adhesive bond between binder and aggregate and
the cohesive forces between binder molecules. Mechanical interlocking
between aggregate particles and that of the asphalt into the aggregate pores
also contributes to the strength and performance of the mixture. The adhe-
sive bond is affected by a number of properties of the asphalt and the
aggregate. Polar molecules present in asphalts such as carboxylic acids,
dicarboxylic anhydrides, sulfoxides, ketones, and pyridinic-type molecules
exhibit strong intermolecular attractions (References 14, 15, and 16).
These molecular types may agglomerate together in asphalt to form micelles
or they may adsorb at the asphalt-aggregate interface. The microscopic
properties of the aggregate such as surface area, porosity, and surface
chemical composition determine the number and nature of active sites for
adsorption of asphalt molecules. Adsorbed material on the aggregate surface
such as dust, ions, and water may prevent the asphalt from bonding to the
aggregate surface. The adsorption of polar molecules at the aggregate
surface results in a molecularly structured layer extending out into the
asphalt binder because of the action of dipole forces induced on asphalt
molecules. The strength of the initial bond and the degree of molecular
structuring around the aggregate may be a significant factor in asphalt-
aggregate mixes that show "tender" behavior and may also be related to the
water resistance of the mix (Reference 9).

Stripping of asphalt molecules from aggregate surfaces occurs by
several mechanisms, including detachment, displacement, emulsification, pore
pressure, and hydraulic scouring. Water damage in asphalt-aggregate mix-
tures may be related to the strength of the bond initially formed between
asphalt and aggregate (Reference 9). Chemical analyses of the fraction of
asphalt strongly adsorbed onto the aggregate both before and after water
treatment have revealed that polar groups may be displaced by the action of
water (Reference 6). Although polar functionalities capable of strong
hydrogen bonding (such as carboxylic acids) are most easily displaced by

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water, strong hydrogen bonding species such as pyridinic-type molecules are thought to be important in increased water resistance of pavement mixtures (References 9, 17, 18).

As an asphalt ages, the concentration of asphaltenes increases due to oxidation and agglomeration of asphalt molecules. Oxidation increases the polarity of asphalt molecules. This leads to increased molecular agglomeration and higher viscosities. Addition of a recycling agent to an aged binder changes the solubility of the binder, altering the properties. Except in the case of 100 percent recycling, recycled pavements use a modifier, virgin asphalt, and new aggregate. Properly recycled binders in mixes are claimed to exhibit better aging characteristics than virgin mixes as shown in Figure 1 (Reference 19). In past years, recycled mixes have been designed by restoring the aged binder to its original viscosity or penetration with a recycling agent. Recently, researchers have developed criteria that make possible the selection of a modifier most chemically compatible with the aged binder to be recycled (Reference 12). The effect that chemical alteration of the aged binder has on the old asphalt-aggregate bond is not known, although 100 percent recycled systems may take months to reach equilibrium (Reference 20). However, in recycling cases where new aggregate is used, the bonding properties of the blend with the new aggregate should be measurably different from the properties of the aged binder new-aggregate bond.

The properties of the aggregate play an important role in determining the properties of an asphalt-aggregate mixture. Aggregates having a silica (SiO_2) content greater than 65 percent are considered "acidic" and generally exhibit greater stripping problems than "basic" aggregates that contain less than 55 percent SiO_2 (Reference 3). Aggregates containing an SiO_2 from 55-65 percent are classified as intermediate. Basic aggregates usually contain considerable amounts of carbonates. Aggregates with high silica contents are more hydrophilic than basic aggregates, primarily because silicates have higher dipole moments than carbonates. Consequently, silica surfaces hold more adsorbed water strongly than do carbonates. The competition of asphalt molecules with water for adsorption sites on the aggregate becomes more intense on silicate surfaces because of the increased resistance of the water to displacement by weaker adsorbing asphalt molecules. The surface area and porosity of the aggregate determine the number and accessibility of

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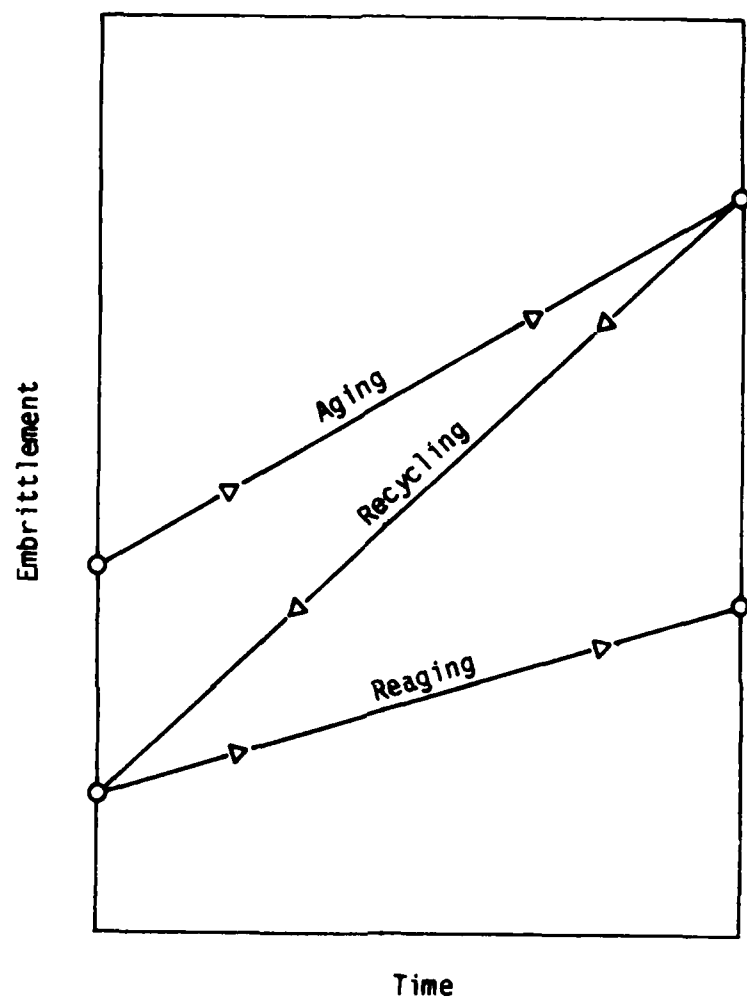
Figure 1. Change in Performance Properties of Recycled Binders (Reference 19).

active sites for adsorbing molecules. An increase in the number of active sites would benefit stripping resistance by requiring more water molecules to displace asphalt molecules from the surface. Surface material - such as dust, water and ions - that is loosely bound reduces the ability of the asphalt to wet the aggregate surface and reduces the number of active sites available for asphalt adsorption. However, adsorbed material on the aggregate surface also reduces the surface energy of the aggregate, thereby, lowering the attraction for water adsorption (References 3 and 21). Although weathered aggregates have more adsorbed material on their surface, they are not as susceptible to water stripping as freshly crushed aggregates (Reference 21).

Several adhesion theories have been postulated for interpreting the properties of asphalt-aggregate mixtures. Mechanical interlocking assumes no chemical interaction between asphalt and aggregate so that all bonding strength is a result of cohesion within the binder and the interlocking of aggregate particles. Thus, the surface texture, porosity, and shape of the aggregate particles determine the bond strength (Reference 3). Chemical bonding theories assume a chemical reaction between the aggregate and the asphalt (Reference 22) although true chemical reactions probably do not occur. However, there are definite formations of weak bonds due to dipole forces.

The molecular orientation theory involves the structuring of asphalt molecules at the asphalt-aggregate interface. The structuring extends out from the surface and into the binder (References 22 and 23). The interfacial theory assumes that adhesion develops because of a decrease in the surface energy of the aggregate as the asphalt is adsorbed onto the surface (References 3 and 22). In reality, the strength of the bond formed between asphalt and aggregate is probably a combination of all the above-mentioned effects. The decrease of surface energy on the aggregate is a result of the formation of weak bonds that induce a dipole on the adsorbed molecules. This dipole effect extends out into the binder matrix causing structuring of the asphalt molecules away from the aggregate surface. The structuring within the binder away from the aggregate surface is probably related to the cohesive strength of the binder.

The following sections present descriptions of the techniques chosen for the study of binders, aggregates, and binder-aggregate mixtures.

B. BINDER ANALYSIS

The following techniques were performed on the binders:

- Modified Clay-Gel Compositional Analysis
- Heithaus Flocculation Ratio
- Viscosity
- Penetration
- Ductility
- Rolling Thin-Film Oven Test (RTFO)

A modification of the Clay-Gel absorption chromatographic method for separating extender oils into generic chemical fractions (American Society for Testing and Materials [ASTM] D2007-75) has been employed extensively for separating asphalt in previous recycling studies (Reference 12). Precipitation of asphaltenes with n-pentane is followed by separation of the remaining components, according to their relative affinities for attapulgus clay and silica gel in a range of polar to nonpolar solvents. Three fractions are obtained from the chromatographic separation: aromatics, polar aromatics, and saturates. The percentages of the four generic chemical fractions have been used as important parameters in seeking the best available modifier for a given aged binder (Reference 12). A considerable base of Clay-Gel data exists on extracted RAP binders, recycling agents, blends, and binders from recycled mixes.

The Heithaus Flocculation ratio method (Reference 24) yields information on the solution properties of asphalts. The technique involves titration of an asphalt sample dissolved in toluene with n-dodecane and determining the amount of titrant necessary to induce flocculation of the asphaltenes present in the sample. The ability of the maltene fraction to disperse the asphaltenes (maltene peptizing power) is a measure of the efficiency of the maltenes to solublize the asphaltenes. The peptizability of the asphaltenes refers to the ease of solublization of the asphaltenes by the maltene fraction. The state of peptization provides a measure of the

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dispersion of the asphaltenes in the maltene fraction. This method has proven to be valuable in determining the compatibility of modifiers with aged asphalt binders (Reference 12). A considerable base of Heithaus data has accumulated from previous recycling studies of Air Force runway pavements (Reference 12). These compatibility data show the effects of recycling agents on the solubility state of the aged binder and the effects of oxidation on a particular binder.

Aged binders and modifiers are subjected to a series of tests to determine their physical properties. Modifiers are tested for weight loss (ASTM D2872), flash point (ASTM D92), and viscosities at 100, 140, and 212 °F (ASTM D2170 and D2171). Viscosity measurements are conducted at these temperatures to determine the temperature susceptibility of the modifier. The modifier viscosity is used to determine the suitability and proportion of the modifier to reconstitute recovered aged binders for a given target consistency. The recovered aged binders, as well as the blends, were tested by a variety of standard methods. Equipment and procedures necessary for conducting these tests are listed in Volume 4.03 of the Annual Book of ASTM Standards (Reference 25).

C. AGGREGATE ANALYSIS

The following tests were performed on the aggregates:

• Surface Area
• Porosity
• X-Ray Fluorescence
• X-Ray Diffraction
• Cation Exchange Capacity (CEC)
• Water-Soluble Ions

The amount of strongly adsorbed components on the aggregate surface is dependent on the surface area of the aggregate (Reference 15). The surface area of the aggregate can be measured using the method developed by Brunauer, Emmett, and Teller (Reference 26). The aggregate sample is cooled to liquid nitrogen temperature and a known quantity of gas (typically nitrogen or krypton) is admitted to the sample chamber. A limited amount of

this gas adsorbs onto the aggregate surface. From the known volume of the sample chamber and several measurements of pressure and temperature as the volume is increased, the volume of gas comprising one adsorbed monolayer may be determined. This volume can be related to the surface area of the sample from the cross-sectional area of the adsorbed gas (Reference 26). Although the surface area that the krypton or nitrogen atom "sees" is different from that of a much larger asphalt molecule, the relative areas between samples of different aggregates is proportional to the amount of strongly adsorbed asphalt molecules (Reference 15).

The porosity of an aggregate may be important in determining the strength of an asphalt-aggregate bond. High porosity results in increased surface area for bonding of more asphalt molecules to the aggregate surface. Also, mechanical interlocking of the asphalt to the aggregate is increased.

Porosity is measured by forcing liquid mercury into the pores of the aggregate under high pressure (up to 33,000 lb/in^2a) and measuring the volume change of the mercury. Assuming a contact angle of 140 degrees and cylindrical pore shape, a distribution of the pore volume as a function of the pore radii is obtained (Reference 27). In larger aggregate particles, internal pores are not accessible to the surface if no connection to the surface is made (closed porosity).

Elemental analysis of bulk aggregate can be performed using X-ray flourescence of samples frozen in lithium fluoride glass (Reference 28) and X-ray diffraction of powdered materials. Further analysis can be made based on weakly adsorbed surface ions desorbed with distilled-deionized water and on exchangeable cations inside the crystal lattice using atomic absorption spectroscopy (Reference 29).

The CEC of soils and minerals is a measure of the number of cations that can be readily exchanged with other cations present in solution (Reference 30). CEC arises from replacement of higher valence cations with lower valence cations of similar size (such as Al^{+3} replacing Si^{+4}) and unequal charge distributions because of imperfections in the lattice structure. These imperfections result in a net charge deficiency in the lattice that is

balanced by adsorbing cations at unbalanced charge centers and crystal faces. These weakly adsorbed cations may be exchanged with other cations present in aqueous solutions causing significant changes in the physical properties of the mineral. CEC has not been widely applied to the study of aggregates although some potential exists for observing CEC effects in asphalt-aggregate mixtures.

The surface free energy of an aggregate is determined by the number of unsatisfied surface bonds that may contribute to the overall charge on the aggregate (Reference 21). Higher surface energy causes more water to be attracted to the surface. Weathering and adsorption of material onto the aggregate surface lower the free energy of the aggregate surface. Thus, weakly adsorbed and easily exchangeable ions may be related to the water susceptibility of the asphalt-aggregate bond. Also, cations such as Fe^{++} and Ca^{++} have been postulated to have effects on the physical and chemical properties of asphalts. Addition of hydrated lime slurries to asphalts and treatment of aggregates with lime reduce the rate of oxidative aging (Reference 31) and the susceptibility of the asphalt-aggregate mixture to water damage (Reference 4).

D. ASPHALT-AGGREGATE MIXTURE ANALYSIS

The following techniques will be applied to asphalt-aggregate mixtures:

- Microcalorimetry
- National Cooperative Highway Research Program (NCHRP) 274 Water Damage Testing
- Indirect Tensile Strength
- Resilient Modulus

Polar functionalities in asphalts have been found to be selectively adsorbed onto aggregate surfaces (References 5, 15, 32, and 33). The types of functionalities found at the interface are carboxylic acids, dicarboxylic anhydrides, sulfoxides, basic nitrogen types, 2-quinolone types, and ketones listed in decreasing order of their relative affinity for aggregate surfaces. Their relative susceptibilities to water damage have been found to decrease in this order although the basic nitrogen types are found to be displaced easier than sulfoxides and 2-quinolone types (References 15 and

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33). These trends are averaged for a variety of common aggregates and may vary slightly depending on the particular aggregate.

Microcalorimetry measures small changes in the heat release of a system such as the heat release which occurs when a solid is dropped into a liquid. The amount of heat released when an insoluble solid is dropped into a pure liquid is proportional to the degree of interaction of the liquid with the solid surface. This interaction results from the strength of the association between solid and liquid or the degree of adhesion between the two phases. This phenomenon is governed by chemical forces such as ionic, dipole-dipole, induced dipole, and dispersion acting at the solid-liquid boundary. Heats of immersion for typical solid-liquid systems involve a sudden release of energy that ceases after a short time (Reference 34).

The heat released by immersion of aggregate into asphalt is unique because the release of energy slowly tapers off but may continue for days. This behavior for asphalt-aggregate systems may be rationalized by assuming that the initial energy released is due to the adsorption of the first monolayer of asphalt molecules surrounding the aggregate resulting in a large initial heat release (see Figure 2, Region A). This initial heat flux is followed by multilayer adsorption because of the induced dipole forces created by the adsorption of the first monolayer (Figure 2, Region B). This multilayer adsorption process is illustrated in Figure 3. The first adsorbed monolayer of polar molecules is furthur polarized by the electric field present at the aggregate surface. This polarization effect is "felt" by molecular neighbors near the monolayer causing further polarization extending out from the aggregate into the binder. This polarization effect causes a molecularly-structured region to form about the aggregate. The strength of the initial bond formed between asphalt and aggregate and the degree of molecular structuring may be related to the water susceptibility and the "tender" or "nontender" characteristics of the asphalt-aggregate mixture (Reference 9).

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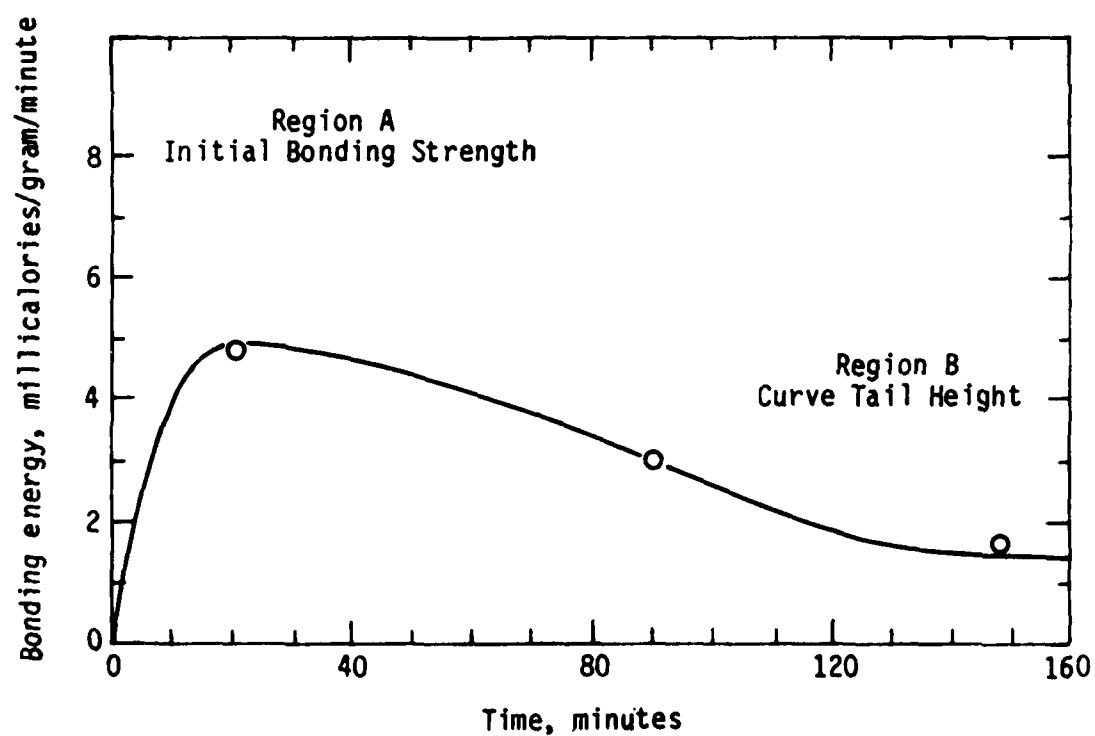
Figure 2. Typical Bonding Energy Curve for a Good Interacting System.
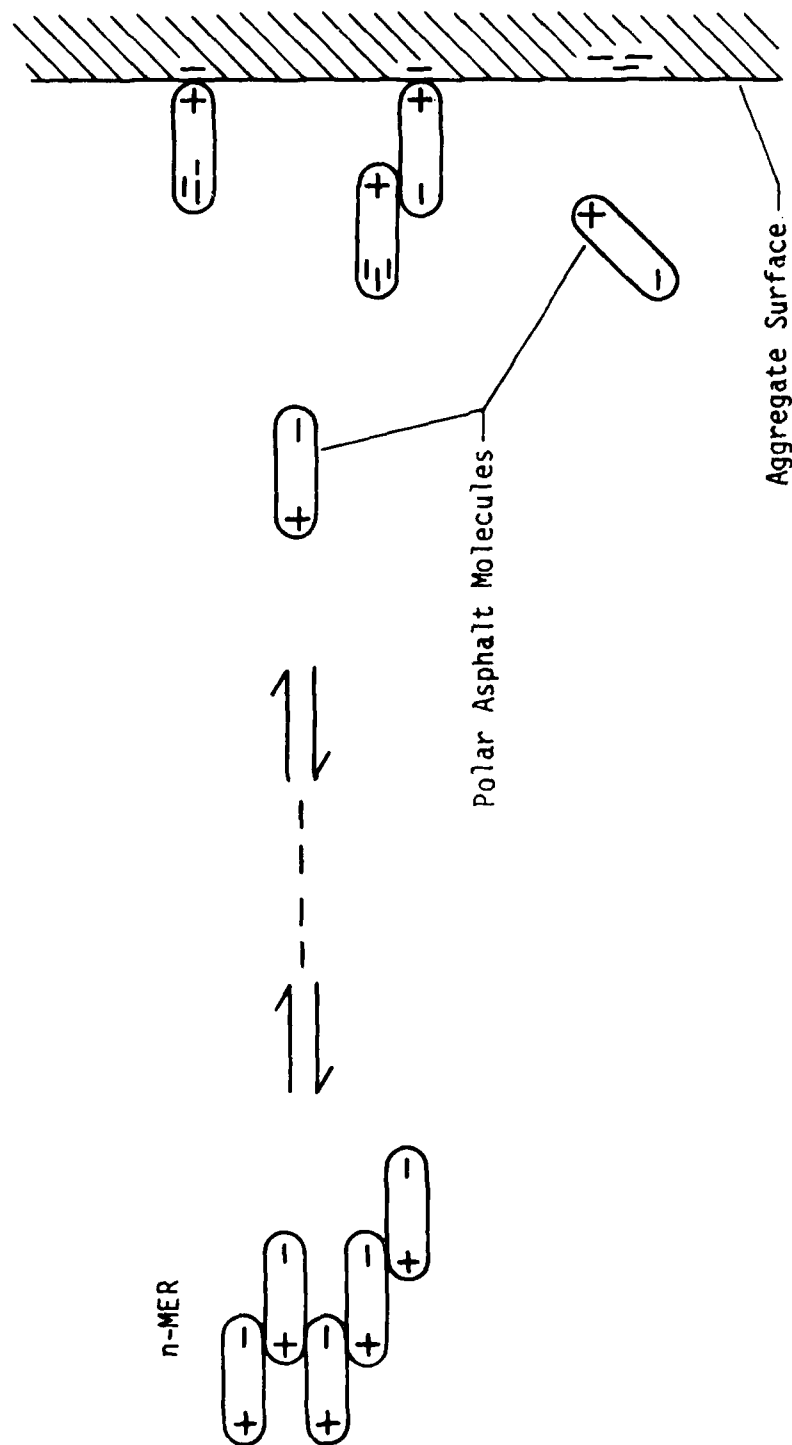
Figure 3. Graphic Representation of Adsorption of Polar Asphalt Molecules at the Aggregate Surface and the Subsequent Multilayer Build-up of Polar Molecules (After Reference 36).

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Microcalorimetry was first applied to the study of asphalt-aggregate interactions by Ensley and Scholz in 1970. The hea⁺ released upon immersion of aggregate into asphalt was postulated to arise from either chemical reaction or from the adsorption of molecules onto the aggregate surface followed by the buildup of subsequent molecular layers (Reference 34). More microcalorimetry experiments (References 35 and 36), flow measurements (References 37 and 38), dipole alignment measurements (References 37 and 38), and contact angle studies (Reference 38) further supported the multilayer adsorption theory. Dipole alignment measurements demonstrated that the more positive region of polar asphalt molecules are aligned towards the aggregate. This is not surprising because of the buildup of net negative charges on aggregate surfaces. Microcalorimetry data have shown that bonding energies increase with temperature. This apparent anomaly is a result of a higher rate of dissociation of asphalt molecular clusters at elevated temperatures, allowing more individual molecules to migrate to the aggregate surface, thus, increasing the bond energy. This phenomenon may have significant effects on the bonding of recycled binder to both old and new aggregate, depending on the characteristics of the hot mixing process (Reference 20).

The relationship between moisture damage and microcalorimetry data is not yet firmly established although some correlations do exist. Curve tail height at some arbitrarily selected time has shown some agreement with stripping test data (Reference 34). A detailed study of the effects of moisture damage on the asphalt-aggregate bond showed no apparent correlation with the degree of water damage as measured by Lottman's procedure (Reference 40) or the water-susceptibility test (Reference 4). However, further work demonstrated that some agreement between the initial bonding strength and Lottman's water damage data is apparent (Reference 9). Excellent correlation between lower curve tail height and tender asphalt-aggregate mixes was noted for pavement mixes with past records of tender behavior (Reference 9).

The NCHRP 274 (Reference 39) is a modification of the Lottman procedure (Reference 40). The test samples are not allowed to reach the maximum water saturation limit and are not exposed to a freeze-thaw cycle as in the Lottman test. Specimens are prepared according to ASTM D4123 to approximately 7 ± 1 percent air void content. Samples are immersed in water for a finite period under 20 inches of mercury vacuum until saturated to the 55-80 percent level. These presaturated specimens are soaked in a 140 °F water bath for 24 hours followed by a 1-hour immersion in a 77 °F water bath. The samples are then tested for indirect tensile strength. Results are reported as the percent retained tensile strength of the dry versus the wet samples. A retained tensile strength of 70 percent or greater is considered to indicate a mixture with good water resistance. Resilient modulus of briquets before and after water treatment was also conducted in this investigation.

The GHD-66 procedure (Reference 41) evaluates mixtures using the Lottman method. This procedure modifies the Lottman tensile strength ratio criteria from a minimum value of 70 to 80 percent. In addition, visual inspection using numerical ratings which range from 0 for nonstripping to 3 for severe stripping is included in the GHD-66 procedure and was also used in this study.

SECTION III
LABORATORY INVESTIGATION AND TEST MATERIALS

A. TEST PLAN

The laboratory investigation was divided into three test plans; general, physico-chemical, and interactive. The test materials included one virgin asphalt, two recycled blends, and two RAP binders. The virgin asphalt was considered to be the control binder. Properties of mixtures made with the control binder were compared to properties made with the blends. Properties of mixtures made with blends were compared with properties from mixtures made with RAP binders.

The general test plan (Table 1) was designed to investigate asphalt-aggregate interaction compatibility by using two extreme performing aggregates where performance is measured by resistance to moisture damage. The Rome aggregate is a nonstripping or slight-stripping aggregate and the Grayson aggregate is the severe stripper. The Nellis (40/60) formula, 40 percent RAP aggregate to 60 percent virgin aggregate, is a neutral aggregage for which no water-damage susceptibility information was available at the time of the study.

The physico-chemical matrix shown in Table 2 is a subset of the general test plan. These tests were planned to determine fundamental physical and surface chemical properties of the aggregate systems. Bulk samples of crushed aggregate were tested for chemical composition, CEC, and water-soluble ions to determine variations from the sieved aggregate portions. Bulk samples were not evaluated for surface area and porosity because material that will not pass through a #4 sieve generally offers negligible contributions. The - #50 + #200 fraction was not tested for chemical properties because variations in composition between the larger fractions are not as large as those of the - #200 fraction. Surface area measurements on each of the sieved fractions were conducted to allow a calculation of the surface area for a given weight of an aggregate mixture. Porosity measurements were conducted on the two fractions used in the microcalorimetry tests (- #4 + #16 and - #16 + #50) and on the - #200 fraction. Porosity measurements of the finer fractions may reveal closed

TABLE 1. GENERAL TEST MATRIX

Binder	Rome limestone	Grayson granite gneiss	Nellis (40/60) formula
Gwinnett AC-30	x	x	x
Nellis blend	x	x	x
Tyndall blend	x	x	x
Nellis RAP	x	x	x
Tyndall RAP	x	x	x

TABLE 2. PHYSICO-CHEMICAL TESTS CONDUCTED ON AGGREGATES

	Surface area	Porosity	Bulk composition by X-Ray fluorescence	Cation exchange capacity	Elemental analysis by atomic absorbtion
Rome bulk sample	-	-	x	x	x
Grayson bulk sample	-	-	x	x	x
- #4 to + #16 sieves	x	x	x	x	x
- #16 to + #50 sieves	x	x	x	x	x
- #50 to + #200 sieves	x	-	-	-	-
- #200 sieve	x	x	x	x	x

Notes: x = Test
        - = No test

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pores accessible only in the - #200 fraction. The results from these measurements were used for explaining mixture property differences between the various asphalt-aggregate combinations.

The interactive tests listed in Table 3 were chosen for testing asphalt-aggregate mixtures. The NCHRP 274 method was applied to triplicate dry and wet specimens of each asphalt-aggregate mixture. The results from the NCHRP 274 test method were used as an indicator of performance. The microcalorimetry test was applied to duplicate mixtures of various asphalt-aggregate combinations. The results from microcalorimetry tests were used to explain the performance differences from the NCHRP 274 measurements on the various asphalt-aggregate combinations. In particular, the comparison between recycled and virgin mixture performance differences, as measured by the NCHRP 274 method, is prime to this study.

B. NOMENCLATURE

The following abreviations are used: GG for Grayson Granite, RL for Rome Limestone, and NF for Nellis (40/60) Formula aggregates. Mixtures are designated by the aggregate symbols followed by the binder used to manufacture the mixture. For example, GG Gwinnett stands for mixtures made with Grayson granite and Gwinnett AC-30 binder.

C. TEST MATERIALS

1. Binders

Test materials were considered from locations where hot recycling was being considered, ongoing or just completed. Project sites identified were Nellis AFB, MacDill AFB, Tyndall AFB, and Hurlburt Field AFB is in a hot-dry region and the others are in hot-wet locations.

The virgin binder used to manufacture mixtures with Rome and Grayson aggregates in previous work (Reference 13) was chosen to be the control asphalt. This asphalt was originally a Lithornia AC-30 asphalt. Gwinnett AC-30 asphalt was substituted because of the unavailability of Lithornia AC-30 at the time the materials were collected. These two asphalts are produced by the same company and used interchangeably in the same state. A comparison of the two materials is presented later. The

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TABLE 3.   INTERACTION TESTS ON ASPHALT-AGGREGATE MIXTURES

Test method	Binder	Rome limestone	Grayson granite gneiss	Nellis (40/60) formula
	Gwinnett AC-30	x	x	x
	Nellis blend	x	x	x
NCHRP 274	Tyndall blend	x	x	x
	Nellis RAP	x	x	x
	Tyndall RAP	x	x	x
	Gwinnett AC-30	x	x	x
Microcal-	Nellis blend	x	x	x
orimetry	Tyndall blend	x	x	x
	Nellis RAP	x	x	x
	Tyndall RAP	x	x	x

Note: x   =   Test

virgin binder is an Amoco product from Gwinnett, Georgia. The source of the crude was not identified and was not a variable in this investigation.

Two aged binders were selected from the RAP samples such that the relative hardness measured by viscosity at 140°F was between 2 and 5. Relative hardness in this study is defined as the ratio of the viscosity of the harder asphalt to the viscosity of the softer asphalt. The aged asphalts selected were from Nellis and Tyndall AFBs.

The RAP from Nellis AFB appeared to be a relatively homogeneous material. The RAP material was collected from a stockpile at various locations in accordance with ASTM sampling procedures. The source pavement feature was initially constructed in 1965. Damaged areas of this pavement feature were replaced with a virgin hot mix in 19__. The RAP was reclaimed and stockpiled by the Las Vegas Paving Company. The binder was extracted and recovered from the Nellis RAP binder and is the harder of the two aged RAP binders.

The Tyndall AFB RAP appeared homogeneous. It was cold-milled from a runway feature. The RAP was sampled from a stockpile in accordance with ASTM sampling procedures. The Tyndall RAP binder was extracted and recovered from the RAP materials. This binder is the softer of the two aged RAP binders.

A modifier satisfying the tentative recycling agent specification (Reference 12) was selected for recompatibilating the two aged binders extracted from the Nellis and Tyndall AFBs. It was assumed that by satisfying the selection criteria, the modifier would be compatible with the two reclaimed binders, which are relatively aged binders of unknown origins. This assumption was later confirmed as shown in the discussion of test results. The modifier (Mobilsol-30) selected was an AR-1000 from Witco Engineering. The MSDS for this product is that which was obtained and evaluated in accordance with ASTM.

Blends prepared with each of the aged binders were adjusted to a viscosity of 3000 poise at 140°F. The amount of the modifier necessary to recycle the aged binders to the specified viscosity were determined independently.

2. Aggregates

Initially, aggregate materials were selected to come from the same sites as the RAP materials. However, virtually no data were available on the past performance record of these aggregates in pavements. Aggregates with well-documented histories of performance were chosen for the experimental design.

A preliminary evaluation of the aggregates from Nellis AFB, MacDill AFB, and Hurlburt Field was conducted to determine aggregate chemical properties. Bulk chemical composition, water displaceable ions, and cation exchange capacity tests were conducted on selected sieve ranges of the naturally screened aggregates. The results indicated variations were significant between sieve ranges for the same aggregate. Also, no data were available on the past performance of these aggregates in paving projects. A severe stripping aggregate and a nonstripping aggregate were selected for the study. A further requirement for these materials was that the total aggregate requirement for a mixture had to come from one source.

The two aggregates selected were from Georgia: a Rome limestone and Grayson granite gneiss. Previous experience with these two aggregates aided in their selection (Reference 13). The Grayson material is known to be a severe stripper and the Rome material a slight stripper. Samples of these aggregates were collected from both Rome and Grayson, Georgia.

The Grayson aggregate originates from an igneous rock system located about 20 miles northeast of Atlanta, Georgia. The parent rock is slightly metamorphosed. The rock deposit consists of contorted and noncontorted phases in its formation. It is highly granular, highly abrasive, and contains about 10 percent pegmatite. The abrasion losses of this deposit are described to exceed the 40 percent maximum which is currently required for most paving operations.

The Rome aggregate comes from a sedimentary rock deposit located about 75 miles northwest of Atlanta, Georgia. This material is described to consist about 98 percent calcite and or dolomite (calcium-magnesium carbonate). The rock is slightly soft and slightly alkali reactive in concrete because of the presence

of a small amount of chalcedony; however, it is accepted for paving operations in the State of Georgia. This aggregate is considered to be a slight stripper in Georgia because the mixtures made with it do not meet the state's minimum retained tensile strength of 80 percent determined by the Lottman procedure (Reference 40). The 80 percent value is a modification of the minimum 70 percent originally established by Lottman.

A third aggregate system was chosen consisting of 40 percent RAP aggregate and 60 percent virgin aggregate. The 40/60 formula was selected to approximate the design used at Nellis AFB to recycle the runway in 1984. The virgin aggregate was obtained from the Lone Mountain pit quarry located about 13 miles northwest of Las Vegas, Nevada and the RAP aggregate was obtained from extracting aged binder from the RAP materials discussed earlier. The Lone Mountain pit quarry was the same source of virgin aggregate for the recycling effort on the runway at Nellis AFB. The RAP used in the recycling effort at Nellis AFB was milled from the same runway.

The Nellis aggregate comes from a dolomitic limestone rock deposit at the Lone Mountain pit quarry. Because of the arid nature of the region, the deposit is located in an area where windblown sand prevails. The sand often shows up in the fine portion of the crushed rock fractions. There was no performance information obtained on this rock with respect to moisture resistance.

The RAP aggregate was obtained by extracting and recovering aged binder from RAP. The aggregate was visually described to consist predominantly of limestone, some quartzite, some rhyolite, and traces of basalt. Preconstruction records were unavailable to locate the original source of this aggregate.

3. Mixtures

Mixtures were prepared using approximately the same grading and binder content (5.0 percent) for the three aggregate systems. The temperature of preparation was 290 ± 10 °F (143 ± 5.5 °C) and the mixtures compacted at 280 ± 10 °F (138 ± 5.5 °C). These temperatures were determined from viscosity measurements of the Gwinnett AC-20 in accordance with ASTM D 1559. The Rome limestone was used to determine the binder content at which a 7 ± 1 percent air void prevailed. These mixtures were prepared at a compaction of 75 blows per face of each briquette.

SECTION IV
MATERIAL TESTING

A. TEST PROCEDURES

The following test procedures were selected for evaluating materials during the course of this research.

1. ASTM C-127, Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate (Reference 25).

2. ASTM C-128, Standard Test Method for Specific Gravity and Absorption of Fine Aggregate (Reference 25).

3. ASTM C-136, Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates (Reference 25).

4. Bulk chemical composition and elemental analyses of aggregates (Reference 29).

5. Surface area and porosity tests on aggregates (References 26 and 27).

6. Modified Clay-Gel Composition Analysis (Reference 13).

7. Heithaus Flocculation Ratio (Reference 13).

8. MIL-STD-620A, Military Test Methods for Bituminous Paving Materials, May 1961 (Reference 42).

9. Air voids analysis by MS-2 procedure (Reference 43).

10. ASTM D-2726, Standard Test Method for Bulk Specific Gravity and Density of Compacted Bituminous Mixtures using Saturated Surface Dry Specimens (Reference 25).

11. ASTM D-4123, Standard Method of Indirect Tension Test for Resilient Modulus of Bituminous Mixtures (Reference 25).

12. Moisture treatment of compacted bituminous mixtures using NCHRP 274 method (Reference 39).

13. Microcalorimetry tests (References 9 and 34).

Other test procedures used in this research are identified in this report at the time of use.

B. TEST DESCRIPTIONS

The test plan outlined the materials and test procedures used. The following discussion will describe each test in detail. The results from these tests are presented in Section V.

1. Physico-chemical Tests on Aggregates

a. Bulk Chemical Composition

Bulk chemical composition of the aggregates was determined by X-ray fluorescence. The aggregate samples were crushed, frozen in lithium fluoride glass, and irradiated with X-radiation. Absorption of the X-rays results in emission of lower-energy radiation that is characteristic of the components present in the aggregate (Reference 28). This technique allows precise determination of the amount of oxides (SiO_x, Al_2O_3, CaO, MgO, for example) and trace metals present in the aggregate sample. The amount of nonadsorbed water [$H_2O(-)$] is determined by baking the aggregate at temperatures from 110-120 °C. Heating the aggregate to 1000 °C allows determination of the amount of adsorbed water on the surface and CO_2 [$H_2O(+)$ + CO_2] released from decomposition of carbonates. The loss on ignition is determined from the [$H_2O + CO_2$] value with a correction for the oxygen liberated upon heating of Fe_2O_3. Ferric oxide (Fe_2O_3) concentrations are determined gravimetrically (Reference 28).

The mineralogic composition of the aggregates was determined by X-ray diffraction of powdered samples. The samples are bombarded with X-rays and the diffraction patterns of the scattered rays recorded. The diffraction pattern is matched against a standard mineral diffraction pattern to determine the composition of the aggregate.

Several sieve ranges were analyzed to determine the variation in composition between the coarse and fine fractions. Bulk specimens from Rome, Grayson, and Las Vegas were tested. Each specimen was ground to pass a #200 sieve prior to bulk analysis.

b. Surface-Adsorbed Ions

The nature of weakly adsorbed surface ions can be determined by washing the aggregate with distilled deionized water and analyzing the wash by atomic absorption spectroscopy. The aggregate sample is immersed in distilled deionized water for 72 hours. The water is then removed and analyzed for ion content by atomic absorption spectroscopy. Preliminary work showed that these ions are indicative of the aggregate composition and that the number of ions detected decreases as the particle size decreases.

c. Cation Exchange Capacity

CEC of soils and minerals is a measure of the number of cations that can be readily exchanged with other cations in an aqueous solution. CEC is measured by leaching cations from the material with neutral pH water: however, carbonate rocks dissolve slightly in neutral pH water yielding false CEC values. To circumvent this problem, aggregates were leached with a solution of barium chloride and triethanolamine ($BaCl_2$/TEA) adjusted to a pH of 8.2 to prevent dissolution of carbonates (Reference 44). The leachate from the aggregates are analyzed by atomic absorption spectroscopy to determine the types and concentration of exchangeable ions. Preliminary studies in Phase I of this work showed that CEC is characteristic of the aggregate type and increases with the surface area of the aggregate.

d. Surface Area and Porosity

The amount of strongly adsorbed asphalt components on the aggregate surface is dependent on the surface area of the aggregate (Reference 6). The surface area of the aggregate was measured, using the method described in References 26 and 27. The surface area measurements were conducted at the University of New Mexico's Powders and Granular Materials

Laboratory on a Quantasorb flow surface area analyzer. Four aggregate sieve ranges were tested on each aggregate.

An aggregate specimen is heated to remove any water that may be adsorbed onto the surface. It is then cooled to liquid nitrogen temperature. A known quantity of nitrogen is admitted to the sample chamber and a limited amount adsorbs onto the aggregate surface. From the known volume of the sample chamber and several measurements of pressure and temperature as the volume is increased, the volume of gas comprising one adsorbed monolayer can be determined. This volume can be related to the surface area of the sample from the cross-sectional area of the adsorbed gas.

Although the surface area that the nitrogen atom "sees" is different from that seen by a much larger asphalt molecule, the relative areas between samples of different aggregates is proportional to the amount of strongly adsorbed asphalt molecules (Reference 6). The surface area of the aggregates measured by this technique includes that of the pores; thus, it is advantageous to measure the porosity of the aggregate.

Porosimetry measurements were conducted on a Quantachrome Autoscan-33 mercury porosimeter. The porosity is determined by immersing the aggregate into mercury and forcing the mercury into the pores with high pressure. The difference in volume of the mercury with and without pressure is related to the pore volume of the aggregate. The amount of mercury forced into the pores as a function of pressure (0 to 33,000 lb/in^2a) yields the pore size distribution of the aggregate.

2.   Physical Tests on Aggregates

Gradation of crushed screenings of virgin and RAP aggregates was conducted following standard ASTM procedures. The Georgia DOT Mix-F grading (Reference 45) was chosen for this study to take advantage of the data base compiled from an earlier study in Reference 13. The results determined the combinations needed to meet the chosen grading for the mixes. Mix-F grading is compared to the AFM-88 grading requirements (Reference 46) in this report.

Specific gravity and water absorption tests were conducted on various fractions of each aggregate in accordance with standard ASTM test procedures listed earlier.

3.   Asphalt and Recycling Agent (Modifier) Tests

A modification of the Clay-Gel absorption chromatographic method (ASTM D2007-80) for separating asphalt into generic chemical fractions has been employed extensively in previous recycling studies reported in Reference 12.   Precipitation of asphaltenes with n-pentane is followed by separation of the remaining components according to their relative affinity for attapulgus clay and silica gel in a range of polar to nonpolar solvents. Three fractions are obtained from the chromatographic separation; aromatics, polar aromatics, and saturates.   The percentages of the four generic chemical fractions have been used as important parameters for selecting the best available modifier for a given aged binder.   A considerable base of Clay-Gel data exists on several reclaimed aged binders extracted from RAP taken from Air Force runways around the country, a number of modifiers used in rejuvenating these binders, and re-extracted binders from recycled mixes. A more complete description of the Clay-Gel technique may be found in Reference 47.

The Heithaus Flocculation Ratio method (Reference 24) yields information on the solution properties of asphalts.   The technique involves titration of an asphalt sample dissolved in toluene with n-dodecane and determination of the amount of titrant necessary to induce flocculation of the asphaltenes present in the sample.   The peptizing power of the maltene fraction required to disperse the asphaltenes, the peptizability of the asphaltenes, and the state of peptization as a whole may be determined from the titration data.   This method has proven to be a valuable technique for determining the compatibility of modifiers with aged asphalt binders.   As with the Clay-Gel method, a considerable base of Heithaus data has accumulated from previous recycling studies of Air Force pavements (Reference 12). A more detailed description of the Heithaus technique is available in Reference 13.

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b. Physical Tests

Aged and virgin recycled asphalt binders and modifiers were
subjected to a variety of tests to determine their physical properties.
Modifiers were tested for weight loss (ASTM D2872), flashpoint (ASTM D92),
and viscosities at 100, 140, and 212 °F. Viscosities were run at these
temperatures to determine the temperature susceptibility of the modifier.
The modifier viscosity at 140 °F was used to determine the proportion
required to reconstitute recovered aged binders to a target viscosity of
3000 ± 600 poises at 140 °F. The recovered aged binders and the blends were
tested by a variety of standard methods. These methods include the
following:

1. Viscosity (ASTM D2170 and D2171) at 140 °F, 212 °F, and
275 °F
2. Penetration (ASTM D5) at 39.2 °F and 77 °F
3. Ductility (ASTM D113) at 60 °F and 77 °F
4. Rolling Thin-Film Oven Test (ASTM D2872)

4. Asphalt-Aggregate Interaction Tests

Microcalorimetry measurements were carried out at the Western
Research Institute in Laramie, Wyoming. A detailed description of the
apparatus will not be presented here, but may be found in Reference 34. A
known weight of aggregate is placed in a cylindrical sample holder from
which it is released and submerged in molten asphalt. The aggregate is
initially separated from the asphalt by a trap door at one end of the
aggregate sample holder. The entire sample cell is placed in the
microcalorimeter and allowed to reach thermal equilibrium at 130 °C for
24 hours. After equilibrium is obtained the trap door is released, allowing
the asphalt and aggregate to interact. The energy released during the
interaction is measured by the thermocouple, amplified, and recorded on a
strip chart recorder.

Bond energy measurements were conducted using the procedure
discussed above on two ranges of screenings. The ranges include material
passing #4 and retained by #16 sieves and material passing #16 and retained

by #50 sieves. Each fraction of each aggregate was tested in duplicate. The two ranges of materials were chosen to determine if the bond energy per gram of material was dependent on the surface area of the aggregate. Microcalorimetry tests are normally conducted on material passing #25 but retained on #48 mesh sizes.

The NCHRP 274 procedure (Reference 39) was used to evaluate mixtures for moisture-damage resistance. In this procedure, test samples are not allowed to reach the maximum water saturation limit and are not exposed to a freeze-thaw cycle as in the Lottman test (Reference 40). Specimens are prepared at approximately 7 ± 1 percent air void content. Specimens are then immersed in water for some time and subjected to approximately 20 inches of head of vacuum until presaturation values of 55 to 80 percent are obtained. The presaturated briquets are soaked in 140 °F water for 24 hours after which they are soaked in a 77 °F water bath for 1 hour and then tested for tensile strength. Results are reported as the percent retained tensile strength of the dry versus the wet samples. A retained tensile strength of 70 percent or greater is considered to be indicative of mixtures with good water resistance using the Lottman criteria.

In this study, mixtures were prepared in accordance with the MIL-STD-620A (Reference 42) using 75 blows per each face of the briquette. Voids were calculated using the MS-2 procedure because air voids determined by the MIL-STD-620A are usually lower because of the use of an apparent specific gravity value in the calculations.

The Rome limestone aggregate and the Gwinnett AC-30 asphalt were used to determine the binder content at which the air void criterion of 7 ± 1 percent was achieved. This level of binder content of 5 percent was established and used throughout the study for all mixtures tested.

SECTION V
RESULTS AND DISCUSSION

A. AGGREGATE ANALYSIS

   1. Chemical/Surface Test Results

   The chemical property data consist of bulk chemical composition, elemental analysis of water-soluble ions and exchangeable bases, and CEC. The bulk chemical composition of each aggregate, water-desorbed ions, and the elemental analysis data on the exchangeable bases and CEC are summarized in Tables on following pages.

   The surface tests consisted of measuring the surface areas and porosities of each aggregate. The surface areas and pore volumes, and the pore volume distribution of the #200 mesh fraction for each aggregate, are summarized as presented. The chemical and surface properties will be discussed in separate sections for each individual aggregate.

   a. Bulk Chemical Composition

   The bulk chemical composition data provide detailed chemical analyses of the aggregate and allows classification of the aggregates according to their composition to be either basic or acidic. The X-ray flourescence technique and wet chemical procedures comprise the main portion of the composition data and were used to determine the percentages of oxides in the aggregates. The precision of these techniques are less than ±0.02 percent. The X-ray diffraction data were matched against mineral standards to provide mineralogical classification of the aggregates. Scanning electron microscopy of the surfaces was conducted to allow visual inspection of the aggregate surfaces. Analyses on the - #4 + #16 mesh range, - #16 + #50 mesh range, - #200 mesh, and bulk rock samples were conducted where possible. Differences in the composition from the coarse to the fine fractions may be caused either by heterogeneity of the sample or environmental factors.

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(1) <u>Grayson</u>. The Grayson, Georgia granite gneiss is known to be a severe stripper by the Georgia DOT and a variety of researchers (References 6, 13, and 48). The aggregate has been widely studied because of its severe stripping characteristics. The composition of this material is given in Table 4. The bulk and sieved fractions are homogeneous in composition with high silica and alumina contents and little carbonate. The high silica content (greater than 65 percent) places this aggretate in the acidic category; thus, it would be likely to exhibit stripping problems. The largest differences in composition between this aggregate and the others studied are the low carbonate and high silica contents. There are also appreciable amounts of alumina not present in other aggregates. This material contains more sodium and potassium oxides than the other aggregates.

(2) <u>Rome</u>. The Rome, Georgia limestone is classed by the Georgia DOT as a slight stripper. The chemical composition of the bulk rock and the crushed fractions are compared in Table 5. The aggregate is a calcitic limestone and contains less than 55 percent silica; thus, it is considered to be a basic rock. A range of 55-65 percent silica content is considered intermediate (Reference 3). The sieved Rome samples are fairly homogeneous with the bulk sample having considerably higher silica (SiO_2) and alumina (Al_2O_3) contents. The sieved samples also have a higher percentage of carbonate. Despite appreciable amounts of silica and alumina, the sample is mainly carbonaceous rock.

(3) <u>Nellis 40/60</u>. The recycling project at Nellis AFB combined 40 percent milled RAP with 60 percent of new aggregate. The new aggregate was taken from the Lone Mountain pit quarry. It was decided that an aggregate blend of 40 percent RAP aggregate plus 60 percent new aggregate would more closely approximate the actual recycling project without seriously deviating from the experimental design for this study. The composition of these materials are given in Tables 6 and 7.

For the Lone Mountain pit aggregate, the composition of the bulk sample and the naturally screened fractions is quite different. The bulk rock sample contains 13.96 percent silica, 18.3 percent MgO, and 22.38 percent

TABLE 4. BULK CHEMICAL COMPOSITION OF THE GRAYSON, GEORGIA AGGREGATE

Constituent	Bulk	- #4 + #16	- #16 + #50	- #200
SiO_2	68.52	71.74	73.17	66.57
TiO_2	0.53	0.31	0.28	0.64
Al_2O_3	15.53	15.13	13.30	14.73
Fe_2O_3	0.89	0.55	1.08	1.19
FeO	1.53	1.57	1.09	0.94
MnO	0.062	0.058	0.065	0.083
MgO	0.564	0.310	0.389	0.642
CaO	1.76	1.53	1.38	3.41
Na_2O	3.78	4.30	3.00	3.74
K_2O	5.85	4.97	5.57	5.16
$H_2O(-)$	0.00	0.08	0.00	0.00
$H_2O(+) + CO_2$	0.46	0.35	0.60	2.08
P_2O_5	0.131	0.056	0.023	0.205
TOTAL	99.60	100.24	100.03	100.02
Total Fe (as Fe_2O_3)	2.59	2.93	2.30	1.60
LOI	0.30	1.91	0.48	0.26
FeO after LOI	0.04	0.12	0.04	0.04

Notes: LOI = Loss on Ignition

Bulk = A piece of rock selected at random from the quarry.

TABLE 5.   BULK CHEMICAL COMPOSITION OF THE ROME, GEORGIA AGGREGATE

Constituent	Bulk	- #4 + #16	- #16 + #50	- #200
SiO_2	26.97	11.14	10.05	12.96
TiO_2	0.17	0.068	0.063	0.13
Al_2O_3	4.56	1.56	1.49	2.85
Fe_2O_3	0.15	0.00	0.03	0.24
FeO	0.98	0.38	0.33	0.47
MnO	0.039	0.037	0.037	0.038
MgO	2.75	2.15	2.00	2.51
CaO	33.90	47.94	46.68	44.09
Na_2O	0.11	<0.01	<0.01	<0.01
K_2O	1.30	0.32	0.30	0.629
$H_2O(-)$	0.04	0.00	0.04	0.00
$H_2O(+) + CO_2$	29.04	36.15	38.92	36.38
P_2O_5	0.133	0.159	0.163	0.181
TOTAL	100.14	99.90	100.1	100.48
Total Fe				
(as Fe_2O_3)	1.24	0.425	0.394	0.758
LOI	28.94	36.11	38.89	36.33
FeO after LOI	0.03	0.04	0.04	0.05

Notes: LOI = Loss on Ignition

Bulk = A piece of rock selected at random from the quarry.

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TABLE 6. BULK CHEMICAL COMPOSITION OF THE LAS VEGAS LONE MOUNTAIN
PIT AGGREGATE

Constituent	Bulk	+ #4	- #200
SiO_2	13.96	7.77	25.40
TiO_2	0.083	0.04	0.27
Al_2O_3	1.54	0.78	4.24
Fe_2O_3	0.61	0.09	1.23
FeO	----	0.08	0.31
MnO	0.043	0.040	0.050
MgO	18.33	19.0	12.9
CaO	22.38	28.1	20.2
Na_2O	0.042	<0.01	0.44
K_2O	0.544	0.37	1.23
$H_2O(-)$	0.21	0.14	1.98
$H_2O(+) + CO_2$	42.15	43.53	31.70
P_2O_5	0.115	0.089	0.09
TOTAL	100.14	100.0	100.0
Total Fe (as Fe_2O_3)	-----	0.18	1.58
LOI	-----	43.52	31.67
FeO after LOI	-----	----	----

Notes: LOI = Loss on Ignition

Bulk = A piece of rock selected at random from the
quarry.

TABLE 7. BULK CHEMICAL COMPOSITION OF THE NELLIS RAP AGGREGATE

Constituent	- #4 + #16	- #16 + #50	- #200
SiO_2	21.46	32.08	30.39
TiO_2	0.18	0.29	0.29
Al_2O_3	4.04	6.21	4.96
Fe_2O_3	0.76	1.03	0.96
FeO	0.28	0.38	0.68
MnO	0.048	0.053	0.048
MgO	10.34	7.30	7.86
CaO	29.80	23.52	24.56
Na_2O	0.83	1.33	0.713
K_2O	1.41	1.93	1.32
$H_2O(-)$	0.00	0.06	0.22
$H_2O(+) + CO_2$	31.20	26.00	27.85
P_2O_5	0.178	0.169	0.150
TOTAL	100.53	100.35	100.0
Total Fe (as Fe_2O_3)	1.07	1.45	1.72
LOI	31.17	25.96	27.78
FeO after LOI	0.05	0.03	0.02

Notes: LOI = Loss on Ignition

CaO. The + #4 fraction has a lower SiO_2 concentration than the - #200 material (7.77 percent versus 25.4 percent), the MgO percentages vary (19.0 percent versus 12.9 percent), the CaO values differ (28.1 percent versus 20.2 percent) as do the loss of ignition percentages (43.53 percent versus 31.70 percent). These compositions place this material in the basic aggregate category. The large differences in chemical composition appear to be due to nonhomogeneity in the samples.

The Nellis RAP aggregate was stripped of the asphalt with a 1:7 methanol/trichloroethylene solvent system. The material is mainly carbonate (MgO + CaO + LOI) with silica concentrations ranging from 21.46 to 30.39 percent; thus, this aggregate is classed as basic. Some concern was expressed over whether strongly adsorbed polar asphalt components might not be removed from the surface of this material by extraction. However, the compositional data available from the tests conducted do not provide any information that proves or disproves the efficiency of the extraction. The assumption is that the aggregate is no different from the other materials studied.

Comparison of the Lone Mountain aggregate and the Nellis RAP aggregate indicates that the two materials are probably from similar sources. In general, the Lone Mountain material contains more carbonates than does the Nellis material. The naturally screened - #200 mesh fraction of the Lone Mountain aggregate may contain windblown dust contributing to the high silicate content.

b. Water Soluble Cations

Aggregate samples washed with distilled deionized water were analyzed for their elemental content by atomic absorbtion spectroscopy. Preliminary studies showed that the water-soluble ions are characteristic of the aggregate type although the ions may not be present in the same ratios found in the bulk rock. The data presented in Table 8 have been normalized to the surface area of the samples to yield a water-soluble ion concentration per area of aggregate.

TABLE 8. WATER SOLUBLE ION CONCENTRATION PER UNIT AREA OF AGGREGATE IN MILLIGRAMS PER SQUARE METER

Aggregate system	Si^{+4}	Al^{+3}	Fe^{++}	Mg^{++}	Ca^{++}	Na^+	K^+	Mn^{++}
Grayson								
- #4 + #16	72.5	20.00	15.75	5.75	8.50	18.13	71.25	<0.63
- #16 + #50	80.0	18.75	11.25	11.00	57.50	12.25	86.25	<0.63
- #200	1.64	0.49	<0.08	0.25	6.72	0.57	1.07	<0.04
Average	51.38	13.08	9.03	5.67	24.4	10.32	52.86	----
Rome								
- #4 + #16	3.37	<0.36	<0.12	2.41	16.14	1.14	1.69	<0.06
- #16 + #50	1.41	<0.23	<0.08	1.72	10.55	0.78	0.84	<0.06
- #200	0.22	<0.07	<0.02	0.47	3.37	0.18	0.13	<0.01
Average	1.67	----	----	1.53	10.02	0.70	0.89	----
Nellis								
- #4 + #16	2.75	<0.28	<0.09	3.21	10.55	1.61	0.61	<0.05
- #16 + #50	3.15	<0.24	<0.08	2.50	9.68	1.90	0.60	<0.04
- #200	0.34	<0.03	<0.01	0.53	2.22	0.28	0.09	<0.01
Average	2.08	----	----	2.08	7.48	1.26	0.43	----

The Grayson aggregate had the highest concentration of ions per area of the three aggregate systems with the water-soluble ion content being an order of magnitude greater than the other aggregates. The concentrations of Si^{+4}, Al^{+3}, Fe^{++}, Na^+, and K^+ ions are significantly higher for the Grayson material. The Rome aggregate is a limestone and is partially soluble in water; thus, the largest concentration of ions found are Ca^{++}. Measureable concentrations of Si^{+4}, Mg^{++}, Na^+, and K^+ ions are also present. As in the Rome limestone, the Nellis 40/60 blend had a large amount of Ca^{++}. The Nellis material has a higher concentration of Mg^{++} as would be expected for a dolomitic limestone. As the surface area increases for each aggregate, the number of water-soluble ions decrease dramatically.

c. Cation Exchange Capacity

The CEC of a material is a measure of the ability to exchange cations with cations present in an aqueous solvent. Previous studies showed the CEC to be characteristic of the material and to increase as the size of the fraction decreases. The CEC data are given in Table 9. As in the water soluble ion concentration per area, the number of ions increases as the mesh size decreases.

The Grayson aggregate has the greater water-soluble ion concentration per area and would be expected to have the largest CEC but it does not. The lower CEC may be somewhat misleading as only Na^+, K^+, Mg^{++}, and Ca^{++} are targeted for analysis. Ions such as Si^{+4}, Al^{+3}, and Fe^{++} are found in appreciable concentrations in a pure water wash of the aggregate and are almost certainly present in the aqueous extract of the CEC procedure.

The Rome limestone has a high CEC for Ca^{++} as would be expected for a calcite but also can exchange considerable amounts of Mg^{++}. The CEC values are highest for the - #200 fraction and decrease for the other fractions. The Rome material also has the highest total CEC values of the other aggregates. The CEC values for Na^+ and K^+ are low; however, the concentrations of the oxides giving rise to these ions in the bulk rock are low.

TABLE 9. CATION EXCHANGE CAPACITY (CEC) OF AGGREGATE SYSTEMS IN UNITS OF MILLIEQUIVALENTS PER GRAM

Aggregate system	K^+	Na^+	Mg^{++}	Ca^{++}	Total CEC
Grayson					
- #4 + #16	0.1	0.0	0.0	0.3	0.4
- #16 + #50	0.1	0.0	0.1	0.4	0.6
- #200	0.8	0.4	0.4	16.2	17.8
bulk	0.1	0.1	0.1	1.1	1.4
Rome					
- #4 + #16	0.0	0.0	0.2	9.1	9.3
- #16 + #50	0.0	0.2	1.0	15.7	16.9
- #200	0.2	0.2	3.3	29.6	33.3
bulk	0.1	0.3	4.3	4.3	9.6
Nellis					
- #4 + #16	0.1	0.1	0.6	7.3	8.1
- #16 + #50	0.1	0.3	1.4	12.5	14.3
- #200	0.5	0.8	8.0	11.7	21.0

Note: Bulk = A piece of rock selected at random from the quarry.

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The Nellis 40/60 material exhibited high Ca^{++} and Mg^{++} CEC values. The CEC values for K^+ and Na^+ reflect the low concentrations of the corresponding oxides found in the whole rock.

d.   Surface Area and Porosity

The surface areas of selected sieve ranges are shown in Table 10 and the pore volume distributions for the - #200 mesh fraction are illustrated in Figure 4. Table 10 also lists values for the total porosity for the - #200 fraction of the aggregate for selected ranges of pore radii.

The Grayson aggregate had the lowest surface area and pore volume. The instrument precision (+ 0.02 m^2/gram) was not great enough to measure a difference in surface area between the Grayson - #4 + #16 and - #16 + #50 fractions. As with the Rome limestone, most of the pore volume was found at the larger pore radii. The Grayson material had more closed pores than the other aggregates as evidenced by the large increase in surface area from the larger to the smaller fractions.

The Rome limestone had intermediate surface area values with an apparent anomaly between the - #16 + #50 and - #50 + #200 fractions; however, this is possibly due to inhomogeneity in sample selection. The material had less pore volume at smaller pore radii than the Nellis aggregate but considerably higher pore volume at the larger radii. The Rome limestone had the largest total pore volume due to the higher porosity at larger pore radii.

The Nellis material had the largest surface area of any of the aggregates tested although the porosity was not the highest. The surface area of the - #200 Nellis material was twice that of the Rome material and seven times that of the Grayson. However, the Nellis material had the largest portion of its surface area residing in pores of 1000 Å radii or less.

A comparison of the pore volume distribution shown in Figure 4 and the total porosities (Table 10) of the aggregates is interesting. The total porosity of each aggregate can be derived from the area under the pore volume distribution curve. Based on the surface area and the pore volume

TABLE 10. SURFACE AREA AND PORE VOLUME CHARACTERISTICS OF THE AGGREGATE SYSTEMS

Aggregate	Gradation ranges			
	- #4 + #16	- #16 + #50	- #50 + #200	- #200
Surface area (m^2 per gram)				
Grayson	0.08	0.08	0.14	1.22
Rome	0.83	1.28	1.13	4.45
Nellis (40/60)	1.09	1.24	1.68	8.88

Pore volumes (cm^3 X 10^{-8})			
	Total	<1000 Å radii	>1000 Å radii
Grayson	105.3	7.7	97.6
Rome	175.1	37.2	137.9
Nellis (40/60)	127.2	51.2	76.0
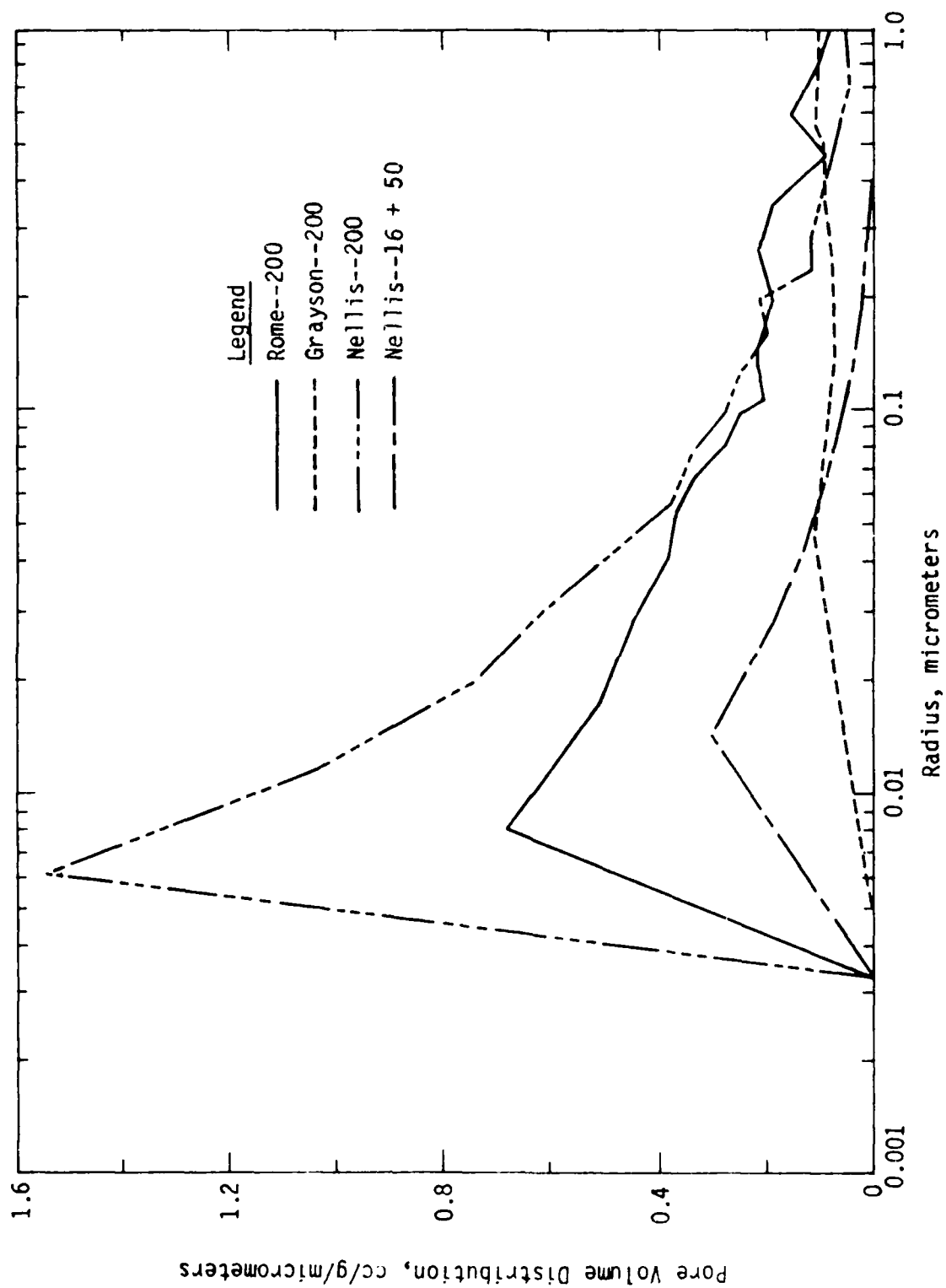
Figure 4.   Pore Volume Distribution for the Material Passing a 200-Mesh Sieve
for each Aggregate as a Function of the Pore Radius.

distribution, the Nellis material would be expected to have the highest total porosity. However, due to the larger pore volume as the pore radius exceeds 1 micron, the Rome limestone has the largest total porosity. At pore radii less than 1 micron, the Nellis material has the highest pore volume.

e.   Summary

Overall, based on the analyses conducted here, the Nellis aggregate would be expected to be the best-performing aggregate. The material has a high surface area because of the increased pore volume at smaller pore radii. This basic aggregate also has the lowest number of ions adsorbed on the surface; thus, the asphalt may adhere to the surface better. The Rome material is also expected to be a satisfactory aggregate because it is basic and contains a moderate surface area. This aggregate also had low amounts of water-soluble ions per area of aggregate and had the highest porosity of the three aggregates studied here. The Grayson aggregate would be expected to be the worst-performing aggregate. The material is acidic in nature, has a low surface area and porosity, and has relatively large amounts of adsorbed material on the surface.

2.   Physical Test Results

a.   Gradation

Table 11 lists gradation test results of crushed screenings for Rome, Grayson, and Lone Mountain aggregate systems. The results for Rome and Grayson aggregates were proportioned to meet the Georgia DOT Mix-F grading which is listed in Table 12. The Lone Mountain test results were used to prepare the Nellis (40/60) formula. This formula consists of 40 percent RAP aggregate and 60 percent Lone Mountain aggregate by weight.

Table 12 also lists the specified grading ranges from the Georgia DOT and the AFM-88-6. The grading values used are generally within the normalized AFM-88-6 limits. Normalization is based on taking the 3/8-inch sieve to be the largest as shown in Table 12.

TABLE 11. GRADATION OF CRUSHED SCREENINGS

Screen, inch	Grayson Limestone		Rome Limestone		Lone Mountain Pit Limestone
	No. 89 stone	No. 810 stone	No. 89 stone	No. 810 stone	3/8-inch stone
1/2	100	100	100	100	100
3/8	100	100	100	100	100
Sieve, #					
4	34	84	29	72	91
8	6	71	5	47	59
16	2	61	2	33	37
30	1	46	1	25	25
50	0.6	30	0.0	18	18
100	0.0	18	0.0	14	14
200	0.0	11	0.0	11	10

TABLE 12. SUMMARY OF GRADATIONS USED IN MIXTURES

Screen, inch	Rome	Grayson	Nellis	GA-DOT-F-MIX	AFM-88-6	[a]AFM-88-6
3/8	100	100	100	90-100	75-93	81-100
Sieve, #						
4	72	68	70	55-75	59-73	63-78
8	46	47	47	44-50	46-60	49-65
16	31	37	34	29-40	34-48	37-52
30	21	28	25	19-34	24-38	26-41
50	14	19	17	12-28	15-27	16-29
100	9	11	10	7-16	8-18	9-19
200	6	6	6	4-7	3-6	3-6

[a]Normalized with respect to the 3/8-inch sieve

b.   Specific Gravity and Water Absorption

Table 13 lists both specific gravity and water absorption
values for the three aggregate systems. The specific gravity values are
within normal ranges for aggregates. The water absorption values suggest that
none of the aggregates can be considered to be absorptive for normal paving
applications. Absorptive aggregates have average absorption values exceeding
2.5 percent.

B.   ASPHALT AND RECYCLING AGENT TEST RESULTS

1.   Chemical Test Results

a.   Clay-Gel Separation Data of Binders

Table 14 summarizes the composition data for the binders used
in this study. The Gwinnett asphalt has a lower saturate content than the
Nellis blend and Tyndall blend binders. However, the Gwinnett asphalt has a
higher asphaltene content than the two blends. The asphaltene content of
Nellis RAP binder was the highest while that of Tyndall RAP binder was almost
equal to the Gwinnett asphaltene content. The general belief is that the
higher the asphaltene content of an asphalt, the higher the viscosity. In
this study, this is true with respect to Nellis RAP binder. However, the
asphaltene contents of Gwinnett and Tyndall RAP binders, were almost equal but
the viscosities were about 1:15 Gwinnett:Tyndall RAP binder. Viscosity
results are discussed later.

The Gwinnett asphalt has a slightly lower polar content but a higher
Polars/Saturates ratio than each of the blends. The lower polar content for
the Gwinnett asphalt suggests that the solvent action in the blends exceeds
that in the Gwinnett. This observation should manifest into lower aging
indexes for the blends than in the Gwinnett asphalt.

The Nellis and Tyndall RAP binders, have lower contents of polar and
aromatic fractions in comparison to both the virgin Gwinnett and blend
binders. This is expected because these fractions are assumed to convert to
the asphaltenes as binder hardening progresses. The action of a compatible

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TABLE 13. SUMMARY OF SPECIFIC GRAVITY OF AGGREGATES

Course Aggregate ASTM C-127							Fine Aggregate ASTM C-128	ASTM D-854
Aggregate size								Filler
- 3/8 inch + #4	- #4 + #8	- #8 + #16	- #16 + #30	- #30 + #50	- #50 + #100	- #100 + #200	- #200	

Grayson granite

	- 3/8 inch + #4	- #4 + #8	- #8 + #16	- #16 + #30	- #30 + #50	- #50 + #100	- #100 + #200	- #200
G_{ga}	2.572	2.624	2.615	2.611	2.627	2.641	2.648	-----
G_{ssd}	2.604	2.638	2.626	2.625	2.643	2.658	2.662	-----
G_{app}	2.656	2.660	2.644	2.646	2.669	2.684	2.686	2.793
$\%_{abs}$	1.235	0.518	0.415	0.508	0.603	0.597	0.535	-----

Rome limestone

	- 3/8 inch + #4	- #4 + #8	- #8 + #16	- #16 + #30	- #30 + #50	- #50 + #100	- #100 + #200	- #200
G_{ga}	2.653	2.647	2.634	2.635	2.615	2.624	2.608	-----
G_{ssd}	2.676	2.672	2.663	2.670	2.658	2.666	2.660	-----
G_{app}	2.716	2.715	2.714	2.430	2.731	2.740	2.751	2.683
$\%_{abs}$		0.934	1.120	1.376	1.617	1.617	1.988	-----

Nellis (40/60) formula

	- 3/8 inch + #4	- #4 + #8	- #8 + #16	- #16 + #30	- #30 + #50	- #50 + #100	- #100 + #200	- #200
G_{ga}	2.678	2.661	2.598	2.568	2.552	2.585	2.580	-----
G_{ssd}	2.708	2.696	2.645	2.622	2.611	2.629	2.635	-----
G_{app}	2.760	2.758	2.727	2.715	2.711	2.706	2.732	2.721
$\%_{abs}$	-----	1.320	-----	2.103	2.306	1.730	2.157	-----

TABLE 14. SUMMARY OF CHEMICAL PROPERTIES OF BINDERS[a]

Chemical property	Gwinnett AC-30		Nellis blend		Tyndall blend		Nellis RAP	Tyndall RAP	AR 1000
	Unaged	Aged	Unaged	Aged	Unaged	Aged			
A. Composition (%)									
Asphaltenes	30.35	33.02	21.33	25.53	20.78	22.76	41.72	30.81	8.61
Saturates	8.97	8.80	13.53	13.09	16.83	16.26	11.31	18.42	14.67
Aromatics	19.04	17.54	18.94	18.96	17.91	17.34	10.86	12.01	25.57
Polars	41.64	470.64	46.20	42.42	44.48	43.64	36.61	38.75	51.15
Polar/Saturate	4.64	4.62	3.41	3.24	2.64	2.68	3.24	2.1	3.49
Asphaltenes + Saturates	39.32	41.82	34.86	38.62	37.61	39.02	52.53	49.23	23.29
Interaction Coefficient (I_c)	1.54	1.39	1.87	1.59	1.66	1.56	0.9	1.03	3.29
B. Solubility									
Asphaltene peptizability, P_a	0.69	0.66	0.49	0.49	0.63	0.62	0.40	0.60	0.81
Maltene peptizability, P_o	1.08	1.14	1.37	1.42	1.18	1.32	1.29	1.16	1.13
State of peptization, P	3.46	3.37	2.67	2.81	3.24	3.42	2.16	2.89	5.96
Limiting dilution ratio Xmin	2.46	2.37	1.67	1.81	2.24	2.42	1.16	1.89	4.96
Limiting titrant volume, T_o	2.44	2.46	1.71	1.83	2.44	23.45	1.20	1.96	5.15
Waxman's cotangent D	2.23	1.91	0.93	0.97	1.64	1.58	0.661	1.45	4.19

[a]RTFO aging system (ASTM D 2872)

NOTE: $I_c = \dfrac{\text{(aromatics + Polars)}}{\text{asphaltenes + Saturates}}$

modifier, MBD-10, after mixing with RAP binders, is observed to: (1) lower
the percent of asphaltenes; (2) increase the polar and aromatic fractions;
(3) increase the Polars/Saturates ratio; and (4) increase the value of the
interaction coefficient, which is desirable.

The interaction coefficient, I_c, is defined* as:

$$I_c = (\text{Aromatics} + \text{Polars})/(\text{Asphaltenes} + \text{Saturates})$$

All these factors are components of the phenomenon of improved
dispersibility. The action of modifier MBD-10 showed by the results
discussed above, satisfies the composition part of the modifier selection
criteria.

b.   Heithaus Solubility Data of Binders

Table 14 lists solubility test results of all binders used.
The Gwinnett asphalt has a higher state of peptization, than the Nellis and
Tyndall blends. This result is in agreement with the higher Polars/Saturates
ratio and the lower saturate content observed in the composition data in
Table 14. However, after RTFO conditioning, the state of peptization for
Gwinnett asphalt decreases slightly, whereas the corresponding state of
peptization for the two blends increases.

The increase in the state of peptization after RTFO
conditioning has previously (Reference 48) been interpreted to mean that the
oxidation species formed during oven aging have enhanced the dispersive action
of the maltenes. A drop in the state of peptization due to oven aging implies
that the formed species enhance the agglomeration of the asphaltenes. Thus,
the recycled blends used in this study experienced an increase in the
dispersive action of the maltenes upon RTFO conditioning. This observation
should lead to lower aging indexes for blends, compared to the Gwinnett virgin
binder, as will been seen when physical data are discussed.

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* From presentation by Mr. Mel Hunter during an asphalt research meeting in
Laramie.

The action of a compatible modifier is seen in improving the dispersive action of the maltene fraction in the aged binder. This improvement can be seen in the state of peptization of the RAP binders compared to the similar values of the blends. Similarly, the values in the remaining parameters, should be higher for the blends than for the RAP binders. These trends were true for the two blends compared to the RAP binders.

2. Physical Test Results

a. Gwinnett/Lithornia AC-30s Compared

Table 15 summarizes the comparative results of the two virgin binders. The physical, composition, and solubility test results suggest that the two asphalts are similar. Physically, the viscosities at 140 and 275 °F are identical. The corresponding Viscosity Temperature Susceptibilities (VTS) computed between these two temperatures are also identical. The remaining physical properties are quite similar.

The trends in both the composition and solubility test results indicate that departures could be partly attributable to test errors. Table 16 shows variability limits determined recently for the composition and solubility parameters from the modified Clay-Gel and Heithaus test procedures.

b. Physical Test Results of all Binders

Table 17 lists physical test results of all binders. The unaged Nellis and Tyndall blends satisfied the AC-30 viscosity requirements specified in ASTM D 3381. The Gwinnett asphalt shows a lower VTS index than the Nellis and Tyndall blend binders. Gwinnett asphalt shows a higher viscosity at 275 °F than the two blends. In fact, the Nellis blend binder shows a lower viscosity at 275 °F than is recommended in ASTM D 3381.

TABLE 15. COMPARING PROPERTIES OF GWINNETT/LITHORNIA AC-30 VIRGIN ASPHALTS

Property	Gwinnett AC-30	Lithonia AC-30
A Physical		
Viscosity, 140 °F,P	3428	3594
Viscosity, 275 °F,cst	575	581
Penetration, 77 °F .1mm	61	72
VTS (140/275) °F	2.46	2.47
B Composition (%)		
Asphaltenes	30.35	27.96
Saturates	8.97	8.61
Aromatics	19.04	22.86
Polars	41.64	40.65
Polars/Saturates	4.64	4.72
Asphaltenes + Saturates	39.32	36.57
C Solubility	unaged	unaged
Asphaltene Peptizability, P_a	0.69	0.67
State of Peptization, P	3.46	3.18
Limiting Dilution X_{min}	2.46	2.18
Maltene Peptizing Power, P_o	1.08	1.06
Limiting Titrant Volume, T_o	2.44	2.09
Waxman's Cotangent D	2.23	2.05

TABLE 16.   SUGGESTED VARIABILITY LIMITS FOR CLAY-GEL/HEITHAUS PARAMETERS
            (REFERENCE 47)

Item	Index	Standard deviation[a]		Coefficient of variation	
		1S	D2S	%1S	%D2S
Composition					
ASP	single operator[b]	0.98	2.77	3.9	10.9
	multiple operator[b]	1.61	4.56	6.1	17.3
SAT	single operator	0.71	2.01	4.2	12.0
	multiple operator	1.56	4.34	8.7	24.6
ARO	single operator	1.16	3.28	6.2	17.4
	multiple operator	3.77	10.66	21.8	61.8
POL	single operator	0.93	2.62	2.5	7.1
	multiple operator	2.30	6.51	6.2	17.4
ASP+SAT	single operator	1.69	4.78	8.1	22.9
	multiple operator	3.17	8.90	14.8	41.9
Solubility					
P_a	single operator	0.03	0.08	3.1	8.7
	multiple operator	0.05	0.13	7.9	22.3
P	single operator	0.16	0.45	6.1	17.2
	multiple operator	0.25	0.79	10.1	28.6
P_0	single operator	0.07	0.19	6.2	17.6
	multiple operator	0.09	0.26	8.7	24.5
X_{min}	single operator	0.17	0.48	9.0	25.4
	multiple operator	0.29	0.79	15.8	44.8
t_0	single operator	0.21	0.59	13.7	38.8
	multiple operator	0.45	1.29	25.6	72.4
Cot Ø	single operator	0.10	0.28	7.3	20.6
	multiple operator	0.21	0.60	13.7	38.8

[a] Standard deviations for composition data in percent
[b] Reference ASTM C670

NOTES: ASP = Asphaltenes T_0 = Limiting titrant volume

         SAT = Saturates   P   = State of peptization

         ARO = Aromatics X_{min} = Limiting dilution ratio

         POL = Polars   Cot Ø = Waxman's cotangent angle

         P_a = Asphaltene peptizability   1S = One standard deviation
                                              (ASTM C670)
         P_0 = Maltene peptizing power

                                         D2S = Maximum acceptable difference
                                               between two results
                                               (ASTM C670)

TABLE 17. SUMMARY OF PHYSICAL PROPERTIES OF BINDERS

Physical property	Binder Designation					
	Gwinnett AMOCO AC-30	Nellis AFB RAP binder	Nellis AFB blend	Tyndall AFB RAP binder	Tyndall AFB blend	AR-1000 modifier
Unaged						
Specific Gravity	1.036	1.044	1.028	1.028	1.023	n/a
Viscosity, 100 °F, P	n/a	n/a	n/a	n/a	n/a	19201
Viscosity, 140 °F,P	3428	197316	2670	51203	2798	519
Viscosity, 212 °F,cst	5504	50227	n/a	20643	n/a	978
Viscosity, 275 °F,cst	575	n/a	288	n/a	370	127
Penetration, 39.2 °F,1mm	23	6	14	12	17	n/a
Penetration, 77 °F,1mm	61	10	51	19	51	143
Ductility, 60 °F cm	100 +	n/a	100 +	n/a	100 +	n/a
Visc-Temp Suspect. (140,275 °F)	2.46	2.82	2.85	2.75	2.66	2.9
RTFO aged						
Viscosity, 140 °F,P	12060	n/a	5522	n/a	5496	962
Penetration, 39.2 °F,1mm	16	n/a	13	n/a	15	n/a
Penetration, 77 °F,1mm	35	n/a	32	n/a	36	89
Ductility, 60 °F cm	79	n/a	95	n/a	22	n/a
Viscosity Ratio	3.52	n/a	2.07	n/a	1.96	1.85
% Weight Loss	n/a	n/a	n/a	n/a	n/a	1.10
% Retained Penetration 39.2 °F	70	n/a	93	n/a	88	n/a
% Retained Penetration 77 °F	57	n/a	63	n/a	71	97

The RTFO aging properties of the virgin asphalt and the blends are also listed in Table 17. The results indicate that the Gwinnett asphalt aged about 2.18 times more than either of the two blends. Retained penetration after RTFO conditioning was higher for the blends than for the virgin asphalt. These results from RTFO aging of the binders confirm the closeup speculations made earlier from the composition and solubility data.

In summary, the choice of a modifier, based on physical and chemical properties listed in the selection criteria in Section IV of this report, suggests the following:

- The oxidized species in the RAP binder are dispersed as measured by the lowering of the asphaltene content; increase in polar and aromatic content; and an increase in the Polars/Saturates ratio of the blend
- The solubility properties of the blend are enhanced compared to those of the RAP binder
- Age hardening defined by the viscosity ratio is improved

Overall, the two sets of results have showed that it is feasible to produce blends equivalent in physical properties to a virgin asphalt. However, the resulting blends do not have composition and solubility properties equivalent to those of a virgin binder. The blends in this study aged at a lower rate than the comparable virgin asphalt. Canessa (Reference 19) made a similar observation in his paper concerning chemical aspects of reconstituting aged pavements in hot recycling. Canessa's observations are shown in Figure 1.

C. MIXTURE TEST RESULTS

1. Compacted Mixture Analysis

Six independently prepared test specimens were made for each asphalt-aggregate mixture. Three of these were tested dry and three were tested wet as discussed in Section III. The results from the testing are discussed in this section.

Analysis of the data proceeded with an Analysis of Variance (ANOVA) from the test results using procedures in References 50 and 51. First, a series of two-way ANOVAs were run on each of the four independent physical tests: dry tensile strength, wet tensile strength, dry resilient modulus, and wet resilient modulus. The purpose of these ANOVAs was to determine the relative effect of different binders and different aggregates upon the strength parameter measured. For all of the strength parameters measured, all three components of a model (aggregate, binder, and binder-aggregate interaction term) were significant. The results of this analysis demonstrated lack of independence between the binder and aggregate.

Concurrent with the ANOVA calculations was a check for homogeneity of variance with the Burr Foster Q test (Reference 50). This check was performed with each ANOVA to ensure the validity of the ANOVAs underlying assumptions of equal variance, and normality of the data. Each check for the homogeneity of variance was satisfied, thus the results of the ANOVAs were interpreted directly.

Since the original ANOVAs indicated a strong interaction term, the analysis proceeded with a series of one-way ANOVAs by aggregate type. This methodology allows the discussion of pertinent aspects of the analysis without the confounding influence of the binder-aggregate interaction term. Along with this analysis, plots of the cell means with corresponding confidence intervals were produced. One set of plots presented the cell mean along with two standard deviations calculated from the individual cells. This set is included in this discussion. The other series of plots; though not shown, consisted of cell means with twice the standard error from the one-way ANOVAs. Finally the cell means were compared by the Newman-Keuls test (Reference 50) to determine statistically significant ranking in the means. The ranking of these cell means is given in Table 18 with the subsequent discussion presenting the highlights of this analysis. The discussion is presented in the following order:

- Grayson Granite (GG) Mixtures
- Rome Limestone (RL) Mixtures
- Nellis (40/60) Formula (NF) Mixtures
- Miscellaneous Test Results
- Model Analysis By Two-Way ANOVAs

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TABLE 18.   NEWMAN-KEULS RANKING OF STRENGTH AND STIFFNESS PROPERTIES

AGG	Binder	DTS	Rank	WTS	Rank	TSR	DRM	Rank	WRM	Rank	RMR
	TR	221.8	A	106.7	A	0.48	695.7	B	665.3	A	0.96
	NR	117.0	D	63.4	C	0.54	1007.3	A	462.3	B	0.46
GG	NB	183.3	B	87.2	B	0.48	481.7	C	446.3	B	0.93
	TB	179.6	B	69.2	C	0.39	421.7	C	222.0	C	0.53
	G	167.0	C	17.3	D	0.10	341.7	C	28.7	D	0.08
	TR	323.7	B	282.3	A	0.87	1435.7	B	1387.7	A	0.97
	NR	407.4	A	219.2	B	0.54	1730.0	A	1050.7	B	0.61
NF	NB	305.7	B	199.5	BC	0.65	920.0	C	543.7	C	0.59
	TB	223.5	C	174.9	CD	0.78	705.0	C	536.3	C	0.76
	G	229.6	C	156.2	D	0.68	609.7	C	363.7	C	0.60
	NR	359.4	A	111.4	C	0.31	1498.7	A	931.0	A	0.62
	TR	255.6	B	182.9	A	0.72	938.3	B	719.7	B	0.77
RL	NB	238.7	B	178.3	A	0.75	666.3	C	709.3	B	1.06
	G	198.9	B	129.7	B	0.65	594.0	C	376.3	C	0.63
	TB	185.7	B	131.4	B	0.71	522.7	C	329.7	C	0.63

Notes: AGG = Aggregate Type

DTS = Dry Tensile Strength

Rank = ranking of means by the Newman-Keuls procedure

WTS = Wet Tensile Strength

TSR = Tensile Strength Ratio

DRM = Dry Resilient Modulus

WRM = Wet Resilient Modulus

RMR = Resilient Modulus Ratio

GG = Grayson Granite

NF = Nellis (40/60) Formula

RL = Rome Limestone

a.   Grayson Granite (GG) Mixtures

(1) Tensile strength. Table 19 lists mixture data for all
binders used with Grayson aggregate. The Grayson mixtures were prepared at
the following mean percent air voids: 7.3 for GG Gwinnett, 6.6 for GG Nellis
blend, 6.7 for GG Tyndall blend, 10.7 for GG Nellis RAP binder, and 6.3 for
GG Tyndall RAP binder. Except for the GG Nellis RAP binder mixture, the
levels of air voids were all within the 7 ±1 percent range. Thus, for the GG
mixtures, except the GG Nellis RAP binder, air voids were considered
constant.

Mixtures were tested at the following mean levels of
percent saturation: 133.1 for GG Gwinnett, 91.7 for GG Nellis blend, 85.7 for
GG Tyndall blend, 81.7 for GG Nellis RAP binder, and 79.1 for GG Tyndall RAP
binder.   The levels of saturation between GG Gwinnett and recycled GG Nellis
blend and GG Tyndall blend were considered to be significantly different. The
GG Gwinnett mixtures were falling apart when they were picked up from the
140 °F hot water bath. However, they firmed up at 77 °F. Thus, recycled
systems with the GG aggregate resisted higher levels of saturation better than
virgin GG Gwinnett mixtures.

Figure 5 shows the mean dry and wet tensile strength of
all GG mixtures along with twice the respective standard deviations. The dry
means of GG Nellis blend and GG Tyndall blend mixtures made with recycled
binders were slightly higher than the mean of virgin GG Gwinnett mixtures.
According to the Newman-Keuls results in Table 18, the difference in the mean
dry tensile strength between recycled mixtures and virgin mixtures shown is
statistically significant and in favor of recycled mixtures.

The GG Nellis RAP binder mixtures had the lowest dry
tensile strength values. This mixture also had the highest percent air voids
of all the GG mixtures. The GG Nellis RAP binder tensile strength values were
significantly lower than similar values for either recycled system. The
GG Tyndall RAP binder mixtures had the highest dry tensile strength of all
GG mixtures presented in Figure 5. The tensile strength of GG Tyndall RAP
binder was significantly higher than any of the values for the recycled
systems. These results concur with the Newman-Keuls results shown in
Table 18.

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TABLE 19. GRAYSON GRANITE MIXTURE RESULTS

Mixture property	G binder	NB binder	TB binder	NR binder	TR binder
Tensile strength					
Dry mean, lb/in^2	167.0	183.3	179.6	117.0	221.8
Error limits[a]	±27.5	±58.4	±14.1	±31.9	±21.7
Wet mean, lb/in^2	17.6	87.2	69.2	63.4	106.7
Error limits	± 3.3	± 5.3	± 6.3	± 4.9	±18.4
Tensile strength ratio, %	10.5	47.5	38.5	54.2	48.1
Resilient Modulus x 10^3 lb/in^2					
Dry mean	342.0	482.0	422.0	1007.0	696.0
Error limits	±45.0	±243.0	±33.0	±267.0	±189.0
Wet mean	30.0	446.0	222.0	462.0	665.0
Error limits	± 1.0	±165.0	±81.0	±134.0	±260.0
Resilient modulus ratio, %	8.8	92.5	52.6	45.9	95.5
Air voids					
Mean, %	7.3	6.6	6.7	10.7	7.0
Error limits	± 0.3	± 0.3	± 0.3	± 0.5	± 0.3
Specific gravity					
Bulk mean	2.301	2.306	2.305	2.245	2.296
Error limits	± 0.007	± 0.006	± 0.007	± 0.012	± 0.007
Theoretical	2.482	2.469	2.472	2.469	
Saturation, %					
Postvacuum mean	76.0	73.3	69.5	74.3	71.4
Error limits	± 8.8	± 7.7	± 4.6	± 5.6	± 9.4
Postwater-treatment mean	133.1	91.7	85.7	81.7	79.1
Error limits	±80.0	± 8.0	± 7.0	± 3.1	± 9.2
Stripping rate[b]	3	3	3	3	3
Vacuum duration,s	5	10	10	10	7.5

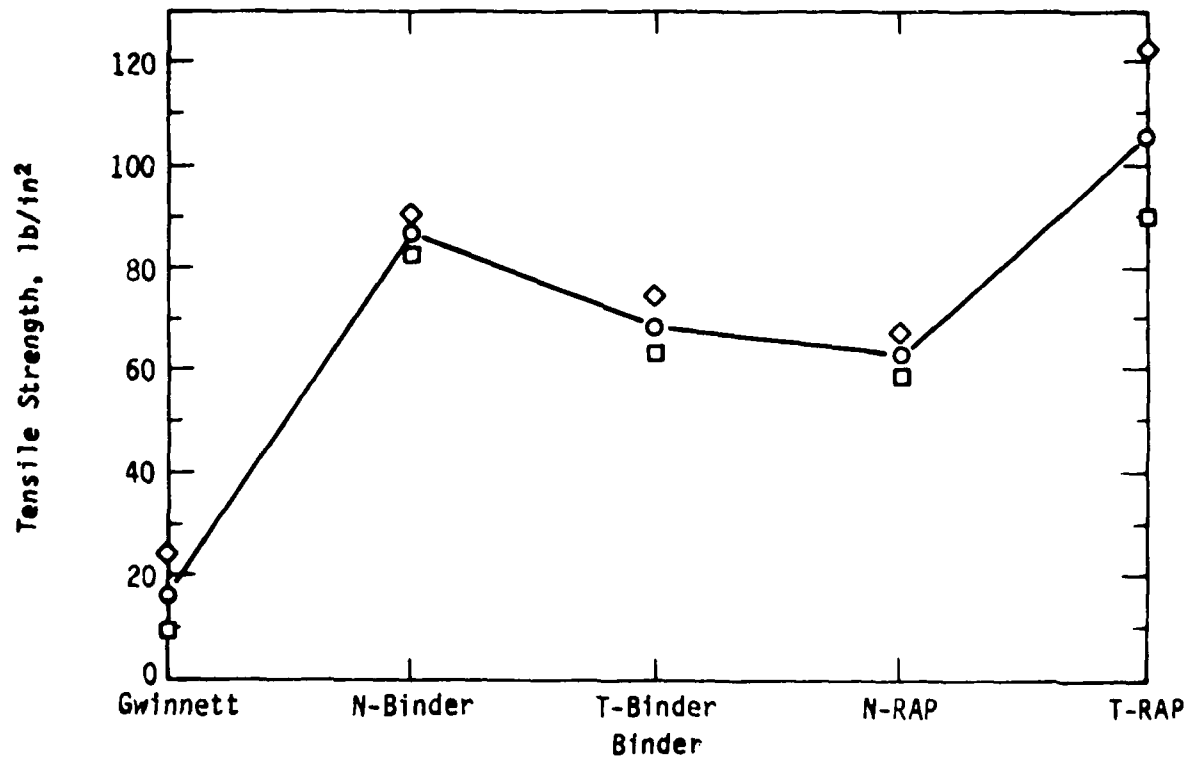[a]Limits given at 95 percent confidence level for all error limits

[b]Numerical ranking of degree of stripping determined by visual observation

NOTES: G = Gwinnett AC-30
        NB = Nellis blend
        TB = Tyndall blend
        NR = Nellis RAP
        TR = Tyndall RAP
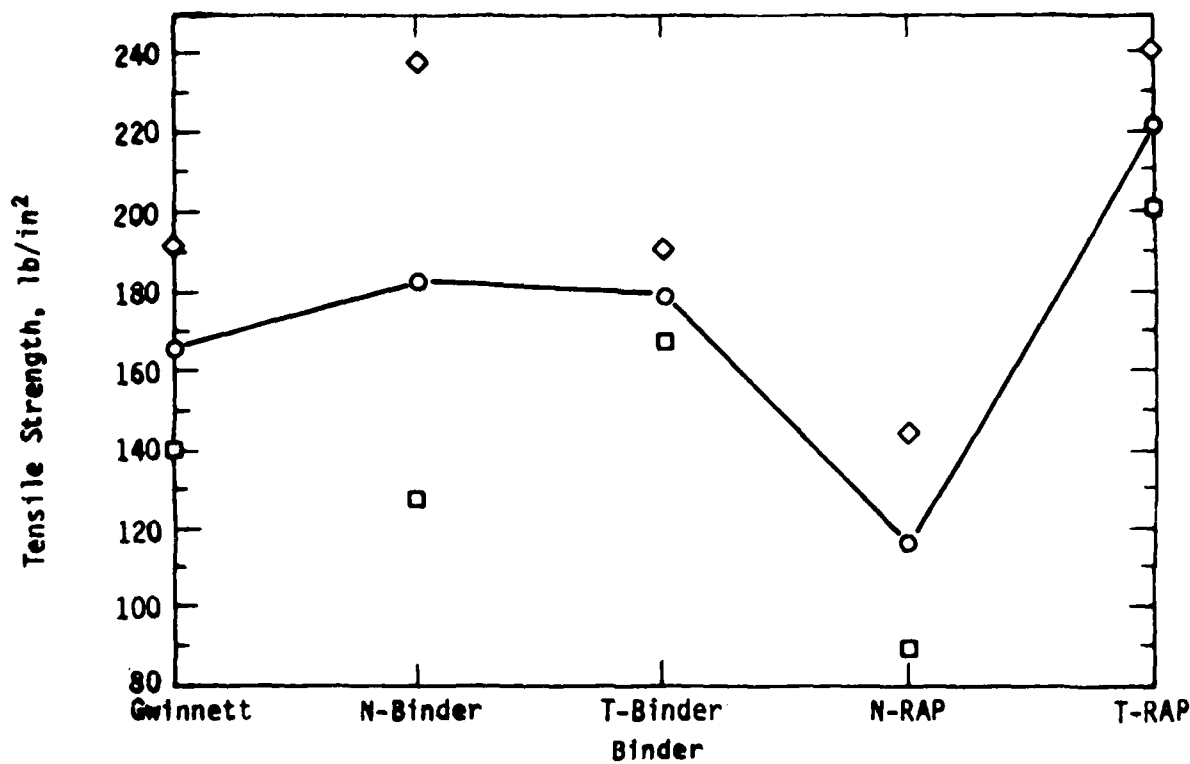
(a) Dry Tensile Strength.

(b) Wet Tensile Strength.

Figure 5. Dry and Wet Tensile Strengths for Grayson Granite Mixtures.

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Wet tensile strength test results are summarized in
Figure 5. GG Nellis blend and GG Tyndall blend mixtures made with recycled
binders had significantly higher tensile strength than did GG Gwinnett
mixtures made with virgin materials after water treatment. GG Nellis RAP
and GG Tyndall RAP binder mixtures had significantly higher tensile strength
than did the virgin GG Gwinnett mixtures after water treatment. However, the
wet tensile strength of GG Nellis RAP and GG Tyndall RAP binder mixtures did
not significantly exceed the wet tensile strength of recycled systems. These
levels of significant differences are inferred from the Newman-Keuls results
in Table 18.

(2) Tensile Strength Ratios. The mean tensile strength ratio
for GG Gwinnett mixtures was 10.5 percent compared to 47.5 and 38.5 percent
for GG Nellis blend and GG Tyndall blend mixtures. These results show the
significant improvement of the GG mixtures by using recycled binders in
comparison to the virgin binder used in this study.

The GG Nellis RAP binder and GG Tyndall RAP binder
mixtures showed tensile strength ratio values of 31.8 and 54.7 percent. These
values are significantly greater than those of the reference GG Gwinnett
virgin mixture system. However, the level of significance of the differences
between the tensile strength ratio values for GG Nellis and GG Tyndall RAP
binder mixtures, and GG Nellis and GG Tyndall blend mixtures was not
determined.

In summary, dry tensile strengths of mixtures made with
recycled binders were statistically higher than the strength of virgin
GG mixtures. Mixed results were obtained when mixtures made with recycled
binders were compared to those made with RAP binders. The wet tensile
strengths of recycled GG mixtures were significantly higher than the wet
tensile strength of virgin GG mixtures. Thus, while the dry strength of the
GG mixtures showed marginal benefits from use of recycled binders, the wet
tensile strength showed marked improvement.

Furthermore, differences between wet tensile strengths of
RAP binder mixtures and recycled mixtures were statistically significant. The
tensile strength ratio values of recycled GG mixtures were significantly

higher than those of virgin GG mixtures. The tensile strength ratio values of the recycled mixtures were comparable to those of RAP binder mixtures for the GG aggregate. Finally, recycled GG mixtures attained lower levels of saturation than the GG virgin mixtures.

(3) Resilient Modulus. Table 19 lists resilient modulus data and Figure 6 shows plots of dry and wet resilient moduli of GG mixtures. In Figure 6, GG Gwinnett, GG Nellis blend, and GG Tyndall blend mixtures show about the same dry stiffness. The ranking by Newman-Keuls analysis in Table 18 showed that these three mixtures were statistically indifferent. The resilient moduli of these three mixtures were significantly lower than the corresponding values for GG Nellis RAP binder and GG Tyndall RAP binder mixtures. The viscosity effect is quite evident in this mechanical property.

In the wet condition, both recycled systems were significantly stiffer than the virgin material system as shown in Figure 6 and the ranking data in Table 18. GG Nellis blend and GG Nellis RAP binder mixtures showed the same wet stiffness and yet the binder viscosities were about 1:74 Nellis blend:Nellis RAP binder. This result violates the significant viscosity effect observed and discussed above for the dry mixtures. Moreover, the GG Tyndall blend had a statistically lower wet resilient modulus value than the GG Tyndall RAP binder mixture. The latter is the norm because the viscosity between Tyndall blend and Tyndall RAP binder is about 1:18.

(4) Resilient Modulus Ratio. The mean resilient moduli ratios were 8.8 percent for GG Gwinnett, 92.5 percent for GG Nellis blend, 52.6 percent for GG Tyndall blend, 45.9 percent for GG Nellis RAP binder, and 71.4 percent for GG Tyndall RAP binder. The differences between the ratio for the virgin mixture GG Gwinnett and recycled mixtures GG Nellis blend and GG Tyndall blend are significant. The differences between the ratios of the recycled mixtures and the RAP binder mixtures may be considered significant for this particular aggregate.

In summary, the dry resilient moduli of GG mixtures with recycled binders were comparable to the corresponding moduli of GG Gwinnett virgin mixtures. The mixtures with RAP binders had significantly higher

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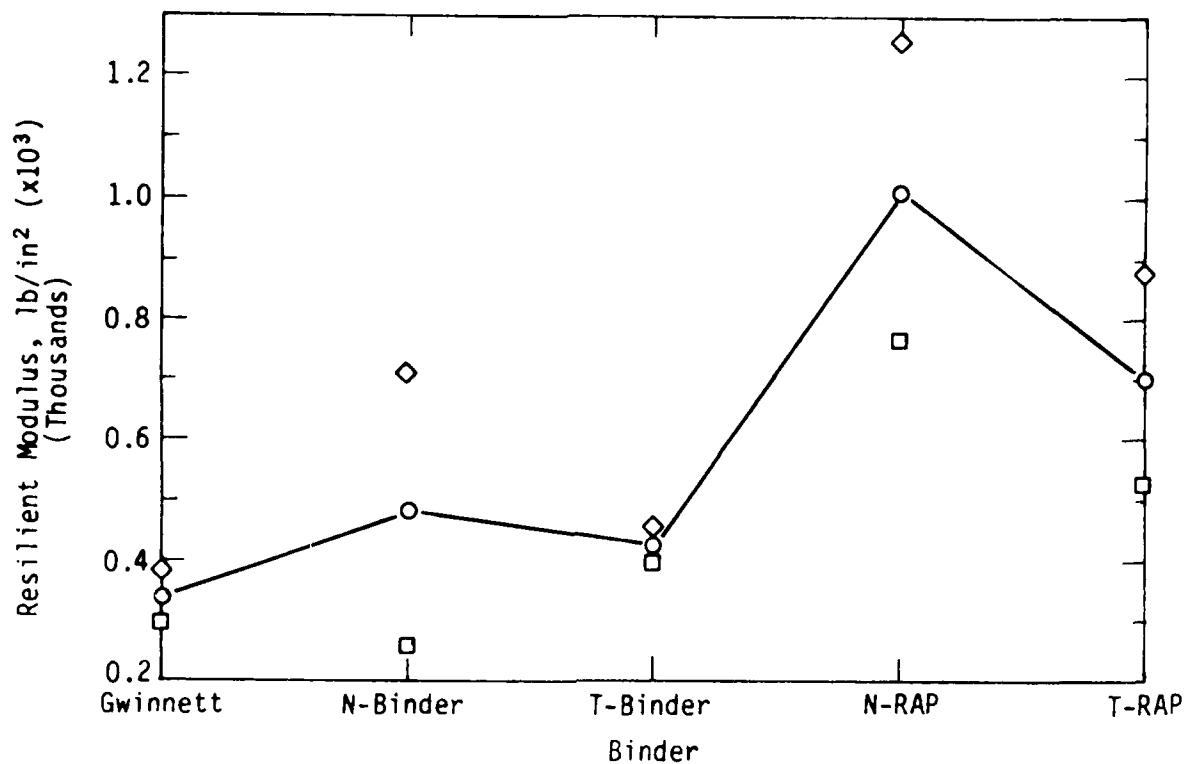
(a) Dry Resilient Modulus.
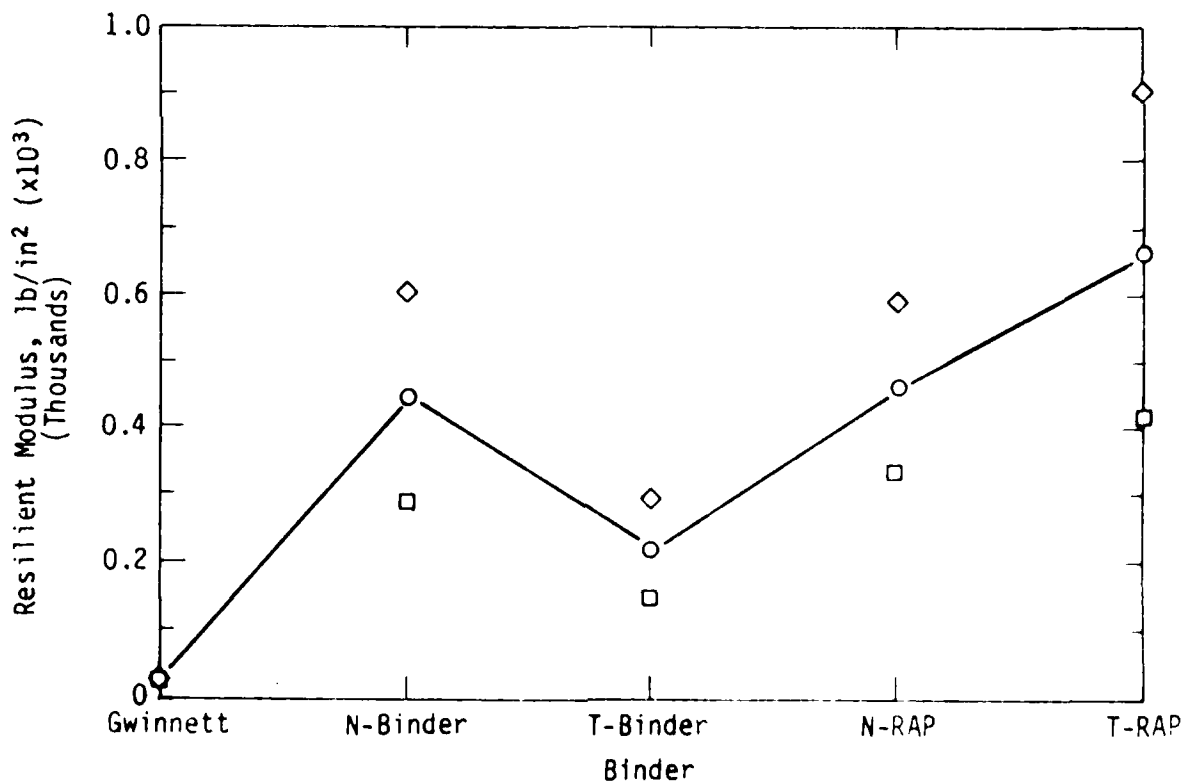
(b) Wet Resilient Modulus.

Figure 6.   Dry and Wet Resilient Moduli for Grayson Granite Mixtures.

moduli values than recycled and virgin mixtures for the GG aggregate system. The binder viscosity effect was pronounced in the dry mixtures.

In wet systems, the resilient moduli of recycled mixtures were significantly higher than those of virgin mixtures for the GG aggregate. The GG Nellis blend recycled system retained the resilient modulus better than the GG Nellis RAP binder mixture. The reverse was observed for the Tyndall blend and Tyndall RAP binder mixtures.

Finally, the resilient modulus ratios of recycled mixtures were significantly higher than those of virgin and RAP binder mixtures.

b.   Rome Limestone (RL) Mixtures

(1) Tensile Strength. Table 20 lists mixture test results for RL aggregate and the five binders. The mixtures were prepared at the following mean percent air voids: 6.1 for RL Gwinnett, 4.2 for RL Nellis blend, 5.9 for RL Tyndall blend, 7.0 for RL Nellis RAP binder, and 6.3 for RL Tyndall RAP binder mixtures. Except for RL Nellis blend and RL Tyndall blend mixtures, the rest of the mixture air voids were within the 7 ± 1 percent limits.

Mean levels of percent saturation for these mixtures were: 74.7 for RL Gwinnett, 111.4 for RL Nellis blend, 73.0 for RL Tyndall blend, 75.0 for RL Nellis RAP binder, and 75.8 for RL Tyndall RAP binder. Mixture RL Nellis blend which had the lowest air voids, had the highest percent saturation. Otherwise, the rest of the mixtures had about the same mean percent saturation.

Figure 7 shows plots of mean dry and wet tensile strengths of RL mixtures. The dry tensile strength of RL Gwinnett virgin mixtures was comparable to the corresponding value of the RL Tyndall blend mixture. There was a statistical difference between the dry tensile strength of recycled RL Nellis blend mixture and the virgin RL Gwinnett mixture. The tensile strengths of RL Nellis RAP binder and RL Tyndall RAP binder mixtures were significantly higher than those of recycled and virgin mixtures.

TABLE 20. ROME LIMESTONE MIXTURE RESULTS

Mixture property	G binder	NB binder	TB binder	NR binder	TR binder
Tensile strength					
Dry mean, lb/in^2	198.9	238.7	185.7	359.5	255.6
Error limits[a]	±37.2	±46.0	±26.6	±144.7	±65.0
Wet mean, lb/in^2	129.7	178.3	131.4	111.4	182.9
Error limits	±39.0	±12.9	±19.8	±14.5	± 6.8
Tensile strength ratio, %	65.2	74.7	70.8	31.0	71.6
Resilient Modulus x 10^3 lb/in^2					
Dry mean	594.0	666.0	523.0	1499.0	938.0
Error limits	±235.0	±117.0	±78.0	±353.0	±253.0
Wet mean	376.0	709.0	330.0	931.0	720.0
Error limits	±154.0	± 48.0	±28.0	±154.0	± 86.0
Resilient modulus ratio, %	63.3	106.5	63.1	62.1	76.8
Air voids					
Mean, %	6.1	4.2	5.9	7.0	6.3
Error limits	± 0.2	± 0.2	± 0.3	± 0.4	± 0.4
Specific gravity					
Bulk mean	2.361	2.363	2.382	2.324	2.348
Error limits	± 0.005	± 0.004	± 0.009	± 0.009	± 0.009
Theoretical	2.515	2.467	2.519	2.500	2.506
Saturation, %					
Postvacuum mean	66.7	101.2	65.4	74.4	65.5
Error limits	± 2.3	± 6.4	± 5.0	± 4.5	± 4.6
Postwater-treatment mean	74.7	111.4	73.0	75.0	75.8
Error limits	± 6.3	± 2.7	± 3.7	±13.8	± 5.1
Stripping rate[b]	2	0	1	0	3
Vacuum duration,s	15	60	10	150	15

[a] Limits given at 95 percent confidence level for error limits
[b] Numerical ranking of degrees of stripping determined by visual observation
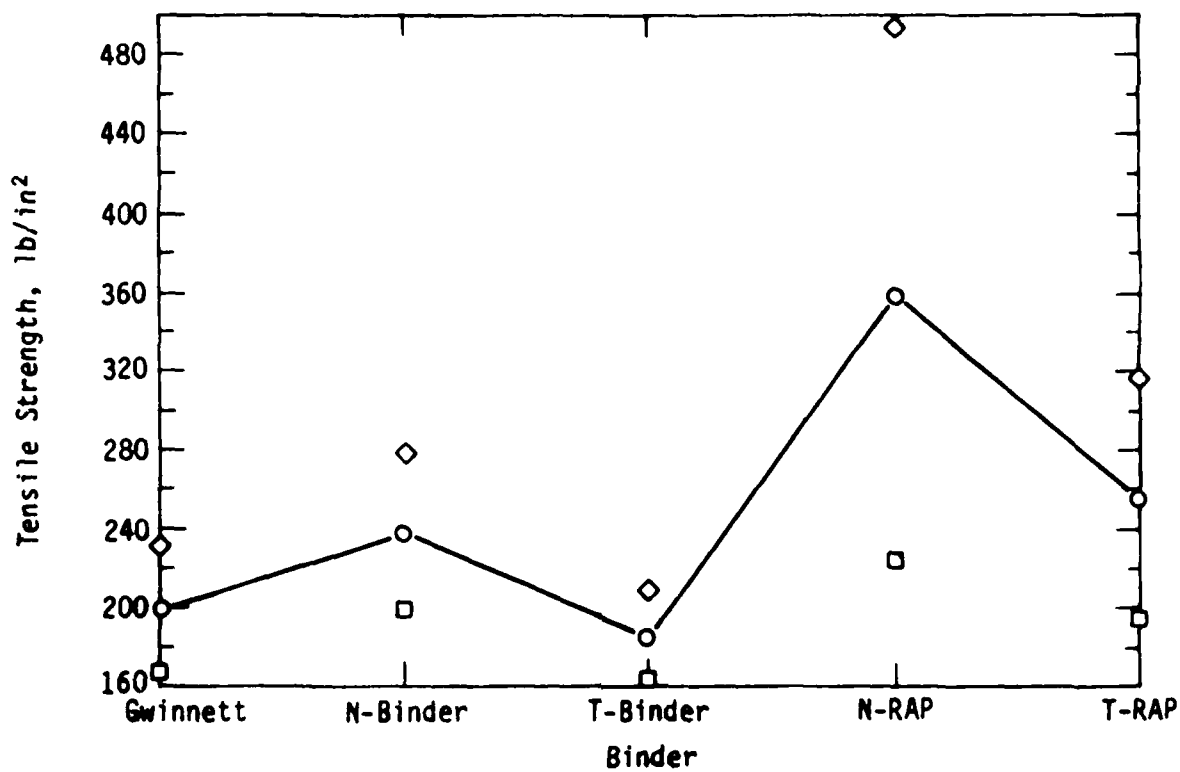
NOTES: G = Gwinnett AC-30
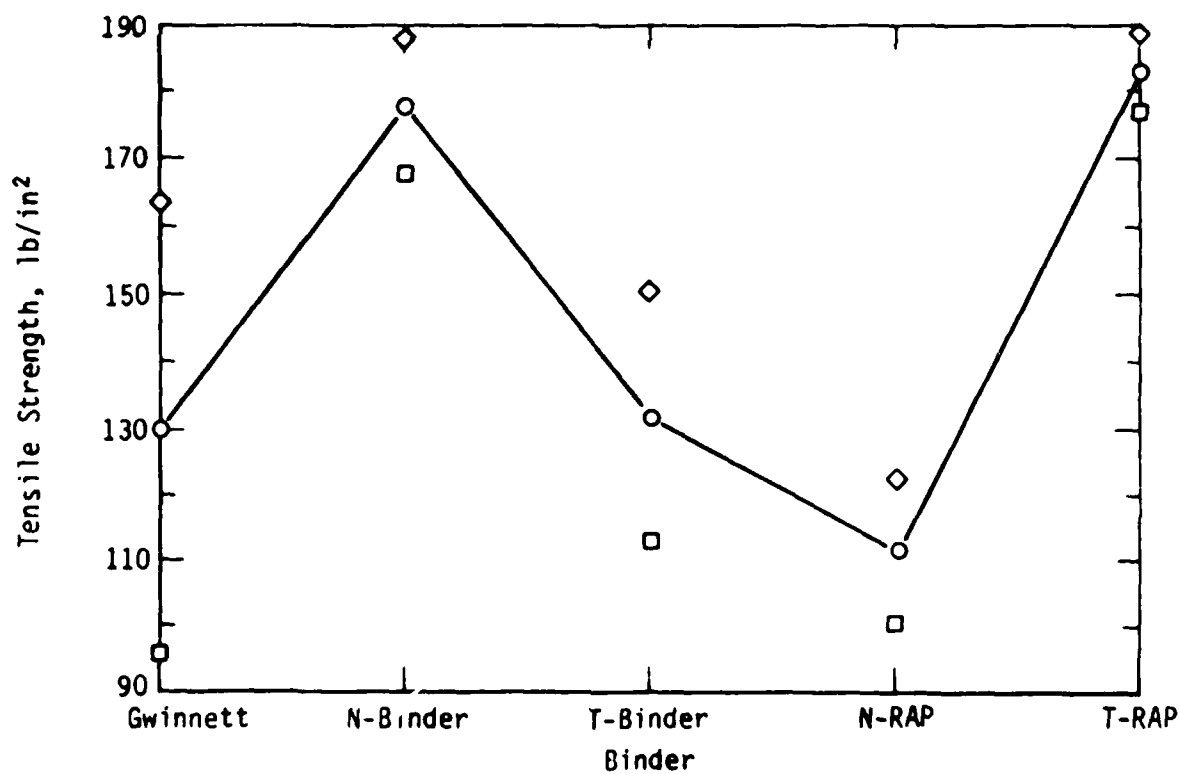   NB = Nellis blend
        TB = Tyndall blend
        NR = Nellis RAP
        TR = Tyndall RAP

(a) Dry Tensile Strength.

(b) Wet Tensile Strength.

Figure 7.   Dry and Wet Tensile Strengths for Rome Limestone Mixures.

The mean wet tensile strength value of the RL Nellis blend mixture was significantly higher than corresponding values of the RL Gwinnett virgin mixture. While the air voids difference did not seem to affect the dry tensile strength, this factor may be considered cause for the difference in the wet tensile strengths. Yet, the RL Nellis blend mixture had significantly higher saturation and lower air voids than the RL Gwinnett mixture which had a higher air void content. The RL Tyndall blend mixture had comparable wet strength to the virgin RL Gwinnett mixture. And the wet tensile strength of RL Nellis blend was significantly lower than that for RL Nellis RAP binder mixtures while the opposite was the result between RL Tyndall blend and RL Tyndall RAP binder mixtures.

(2) <u>Tensile Strength Ratios</u>. The mean tensile strength ratios for RL mixtures in percent were: 65.2 for RL Gwinnett, 74.7 for RL Nellis blend, 70.8 for RL Tyndall blend, 31.0 for RL Nellis RAP binder, and 71.6 for RL Tyndall RAP binder. The tensile strength ratios for recycled mixtures are higher than those of the control virgin mixture. The RL Tyndall RAP binder mixture had a tensile strength ratio value comparable to the recycled mixtures while the RL Nellis RAP binder mixture had the lowest tensile strength ratio value of all mixtures made with RL aggregate.

Tensile strength ratio values are sometimes misleading because a system with a lower value could have a higher wet tensile strength than another system with a high tensile strength ratio value. Likewise, two systems could have about the same tensile strength ratio value but have totally different tensile strength values. An example of the first type is that of the RL Nellis RAP binder mixture (Table 20) with a mean wet tensile strength of 111.4 lb/in^2 and a tensile strength ratio of 31 percent; and the GG Nellis RAP binder mixture (Table 19) with a mean wet tensile strength of 63.4 lb/in^2 and a tensile strength ratio of 54.2 percent. These two systems used the same binder. An example of the second type is that of RL Gwinnett with a tensile strength ratio of 65.2 percent and a mean wet tensile strength of 129.7 lb/in^2, and NF Gwinnett (Nellis Formula aggregate with Gwinnett asphalt) having a tensile strength ratio of 68.0 percent and a mean wet tensile strength of 156.2 lb/in^2.

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This discussion implies that the rejection of a mixture on the basis of tensile strength ratio should be supported by values of wet tensile strength, otherwise tensile stress limits need to b. developed.

In summary, the dry tensile strengths of recycled mixtures using the RL aggregate were partly comparable to that of virgin materials. For the RL Nellis blend mixture, the difference in dry tensile strength compared to that of the virgin mixture was statistically significant. The corresponding tensile strength ratio values of recycled systems were higher than that of the virgin mixture. RL Nellis RAP binder and RL Tyndall RAP binder mixtures had significantly higher dry tensile strength than recycled mixtures.

The results comparing wet tensile strengths between the recycled mixtures and RAP binder mixtures were mixed. The wet tensile strength of RL Nellis blend was significantly higher than that of RL Nellis RAP binder, however, the wet tensile strength of RL Tyndall blend was statistically lower than that of RL Tyndall RAP binder. Finally, the need for developing criteria using tensile strength ratio along with wet tensile strength for evaluating the resistance of bituminous mixtures to the action of water is implied from the results of this analysis.

(3) Resilient Modulus. Results of dry and wet resilient moduli for the RL mixtures are listed in Table 20 and the means are plotted in Figure 8. Dry resilient moduli values for RL Gwinnett virgin mixtures are comparable to those of the two recycled mixtures. Newman-Keuls ranking for the three mixtures was identical.

The mixtures prepared with Nellis RAP binder had significantly higher dry resilient moduli than the recycled and virgin mixtures for this aggregate. The mixture with Tyndall RAP binder was also significantly stiffer than the recycled and virgin mixtures. Thus, the viscosity effect which was reported earlier is evident.

In Figure 8, mean wet resilient modulus for RL Tyndall blend mixture is comparable to that of the RL Gwinnett virgin mixture. However, the RL Gwinnett virgin mixture was significantly less stiff than
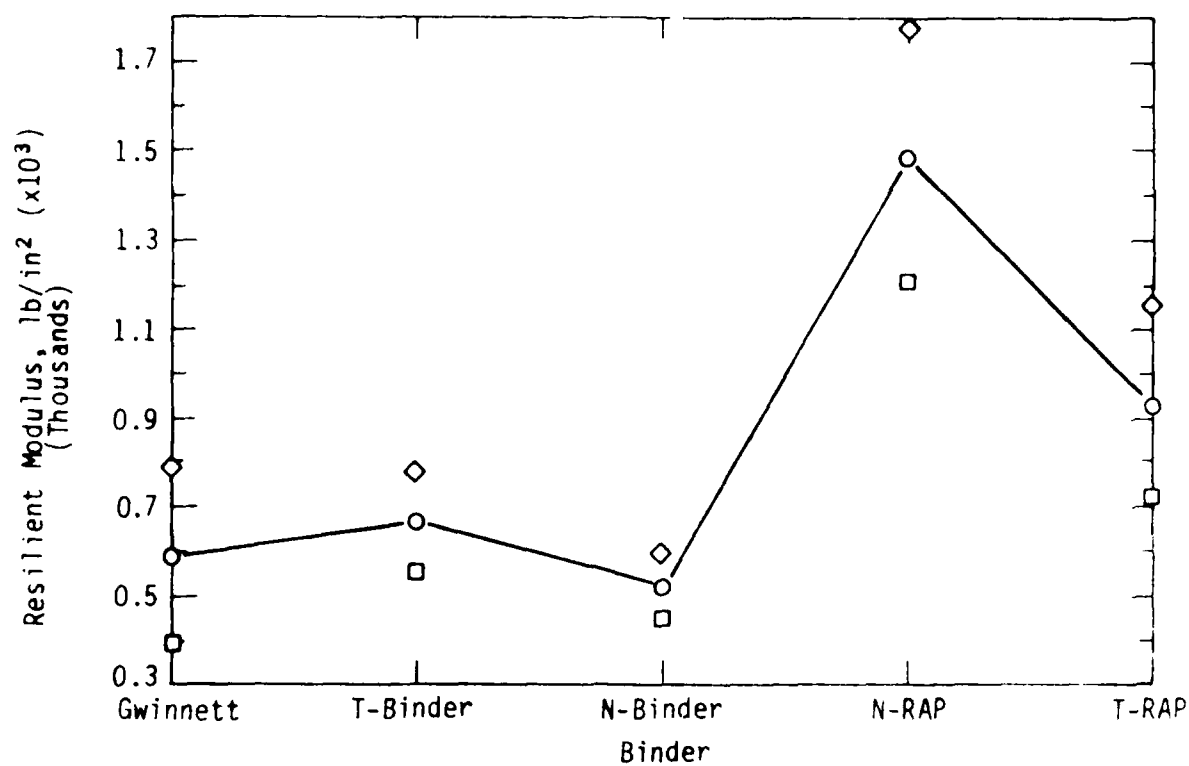
(a) Dry Resilient Modulus.
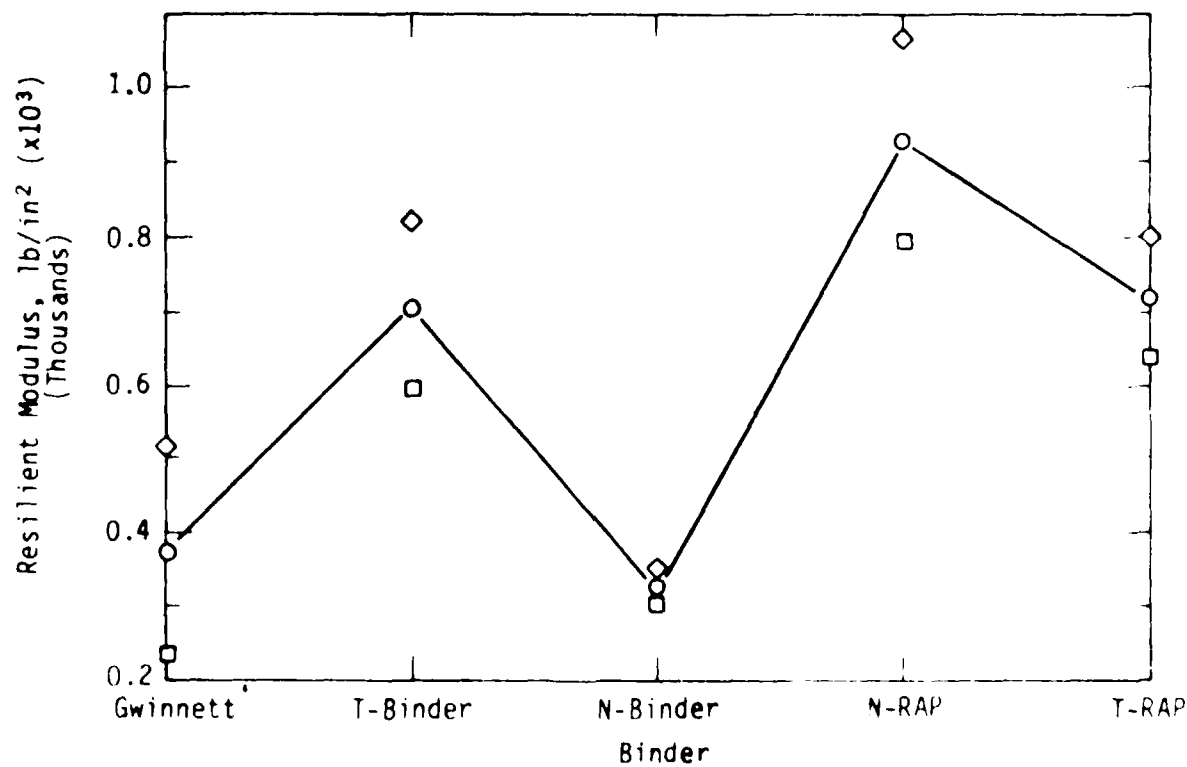
(b) Wet Resilient Modulus.

Figure 8. Dry and Wet Resilient Moduli for Rome Limestone Mixtures.

the RL Nellis blend recycled mixture. The difference in stiffness may be due to the difference in air voids. Mixtures made with RAP binders were significantly stiffer than the corresponding recycled mixture. Thus, the viscosity effect was still evident.

(4) Resilient Modulus Ratios. Resilient modulus ratios for the mixtures in percent were: 63.3 for RL Gwinnett, 106.5 for RL Nellis blend, 63.1 for RL Tyndall blend, 62.1 for RL Nellis RAP binder, and 76.8 for RL Tyndall RAP binder. With the exception of the RL Gwinnett mixture, these ratios are not significantly different from one another.

In summary, the stiffness of recycled mixtures was not statistically higher than the stiffness of virgin mixtures for the RL aggregate. The RAP binder mixtures had significantly higher dry resilient moduli than the recycled and virgin mixtures. This result was attributed to the viscosity effect. For the wet systems, one recycled system had a statistically higher resilient modulus than the virgin system, and another recycled system was of comparable resilient modulus. The retained resilient moduli values were equally comparable, though one system had a high value of 106.5 percent.

In conjunction with using the resilient modulus ratio, wet resilient modulus should be considered in judging the water susceptibility of mixtures. A range of 100 - 1,500 k/in^2 for resilient modulus is usually considered adequate for paving operations. Thus, for recycled mixtures, a range for wet resilient moduli of 100 - 1,000 k/in^2 should be considered along with the modulus ratio. Wet resilient moduli for the recycled systems in this study ranged from 222 to 710 k/in^2 and the ratio varied from 0.53 - 1.06.

c. Nellis (40/60) Formula (NF) Mixtures

(1) Tensile strength. Test results for dry and wet NF mixture properties are summarized in Table 21. The NF mixtures were made at mean percent air voids of 4.7 for NF Gwinnett, 4.2 for NF Nellis blend, 5.9 for NF Tyndall blend, 7.0 for NF Nellis RAP binder, and 6.3 for NF Tyndall RAP binder. This mixture system had more variability with air voids than the previous two systems discussed earlier. The mixtures were tested at mean

TABLE 21. NELLIS (40/60) FORMULA MIXTURE RESULTS

Mixture property	G binder	NB binder	TB binder	NR binder	TR binder
Tensile strength					
Dry mean, lb/in^2	229.6	305.7	223.5	440.8	323.7
Error limits[a]	±27.2	±69.4	±34.9	± 37.8	±14.7
Wet mean, lb/in^2	156.2	199.5	174.9	219.2	282.3
Error limits	±31.6	±62.1	± 6.8	±68.5	±13.5
Tensile strength ratio, %	68.0	65.3	78.4	49.7	87.2
Resilient Modulus x 10^3 lb/in^2					
Dry mean	610.0	920.0	705.0	1730.0	1436.0
Error limits	±135.0	±414.0	±312.0	±789.0	± 53.0
Wet mean	364.0	544.0	536.0	1051.0	1388.0
Error limits	± 98.0	±314.0	±216.0	± 69.0	±579.0
Resilient modulus ratio, %	59.7	59.1	76.1	60.8	96.7
Air voids					
Mean, %	4.7	4.6	5.5	7.7	4.7
Error limits	± 0.7	± 0.4	± 0.7	± 0.8	± 0.3
Specific gravity					
Bulk mean	2.389	2.399	2.380	2.352	2.392
Error limits	± 0.042	± 0.024	± 0.000	± 0.008	± 0.009
Theoretical	2.507	2.514	2.512	2.550	2.510
Saturation, %					
Postvacuum mean	67.7	51.9	62.1	56.2	49.8
Error limits	±16.7	±31.1	± 6.9	±19.8	±11.6
Postwater treatment mean	72.7	53.8	65.4	53.0	61.3
Error limits	±13.7	±29.8	±10.0	±15.2	±12.9
Stripping rate[b]	3	3	3	3	0
Vacuum duration,s	5	2400	25	840	10

[a] Limits given at 95 percent confidence level for all error limits
[b] Numerical ranking of degrees of stripping determined by visual observation

NOTES: G = Gwinnett AC-30
       NB = Nellis blend
       TB = Tyndall blend
       NR = Nellis RAP
       TR = Tyndall RAP

percent saturation of 72.7 for NF Gwinnett, 53.8 for NF Nellis blend, 65.4 for NF Tyndall blend, and 58.0 for NF Tyndall RAP binder. The saturation values were not significantly different.
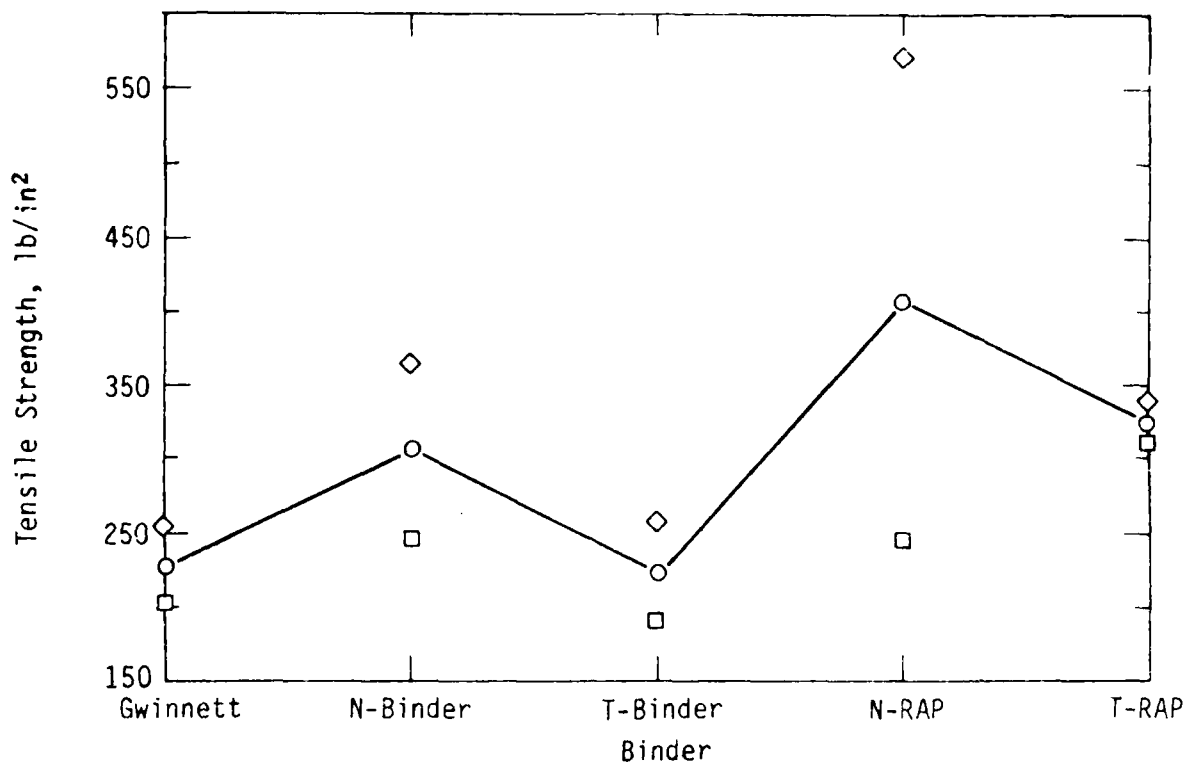
Figure 9 shows plots of dry and wet tensile strength of NF mixtures. The mean dry tensile strength for the NF Gwinnett virgin mixture was comparable to that of the NF Tyndall blend recycled mixture but statistically lower than that of the NF Nellis blend recycled mixture. The dry strength of NF Tyndall RAP binder and NF Nellis RAP binder was significantly higher than the dry strengths of both recycled and virgin NF mixtures. These observations concur with the Newman-Keul rankings in Table 18.

The wet tensile strength shown in Figure 9 and the Newman-Keuls rankings in Table 18 indicate that the strengths of recycled mixtures are statistically higher than the strength of the control virgin mixture. There are significant differences between wet tensile strength for each mixture made with RAP binder and the corresponding recycled mixture.
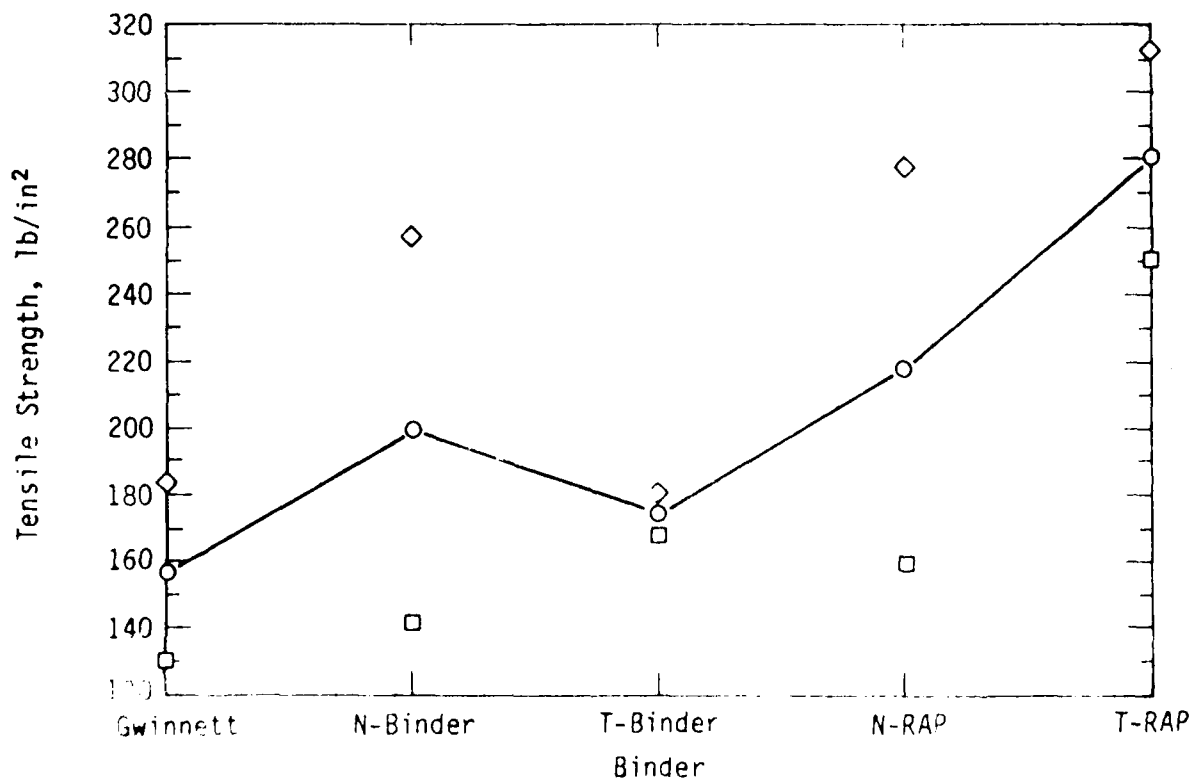
(2) Tensile Strength Ratios. Mean tensile strength ratio values in percent were 68.0 for NF Gwinnett, 65.3 for NF Nellis blend, 78.4 for NF Tyndall blend, 49.7 for NF Nellis RAP binder, and 87.2 for NF Tyndall RAP binder. The ratios for the NF Gwinnett and the recycled systems are not significantly different. The ratios between the RAP binder mixtures may be considered significantly different. However, the NF Tyndall RAP binder mixture had a higher tensile strength ratio value than any of the mixtures for this aggregate.

In summary, regardless of the variability in air voids, the dry and wet tensile strengths for the recycled mixtures were statistically higher than those of the reference virgin mixture. Mixtures made with RAP binders had significantly higher dry and wet tensile strengths than did the rest of the mixtures.

Recycled NF mixtures had comparable tensile strength ratio values to that of the virgin mixture. This suggests that the NF aggregate results were not asphalt specific. The NF Tyndall RAP binder mixture had a higher tensile strength ratio value than any other NF mixture and NF Nellis RAP binder had the lowest tensile strength ratio value.

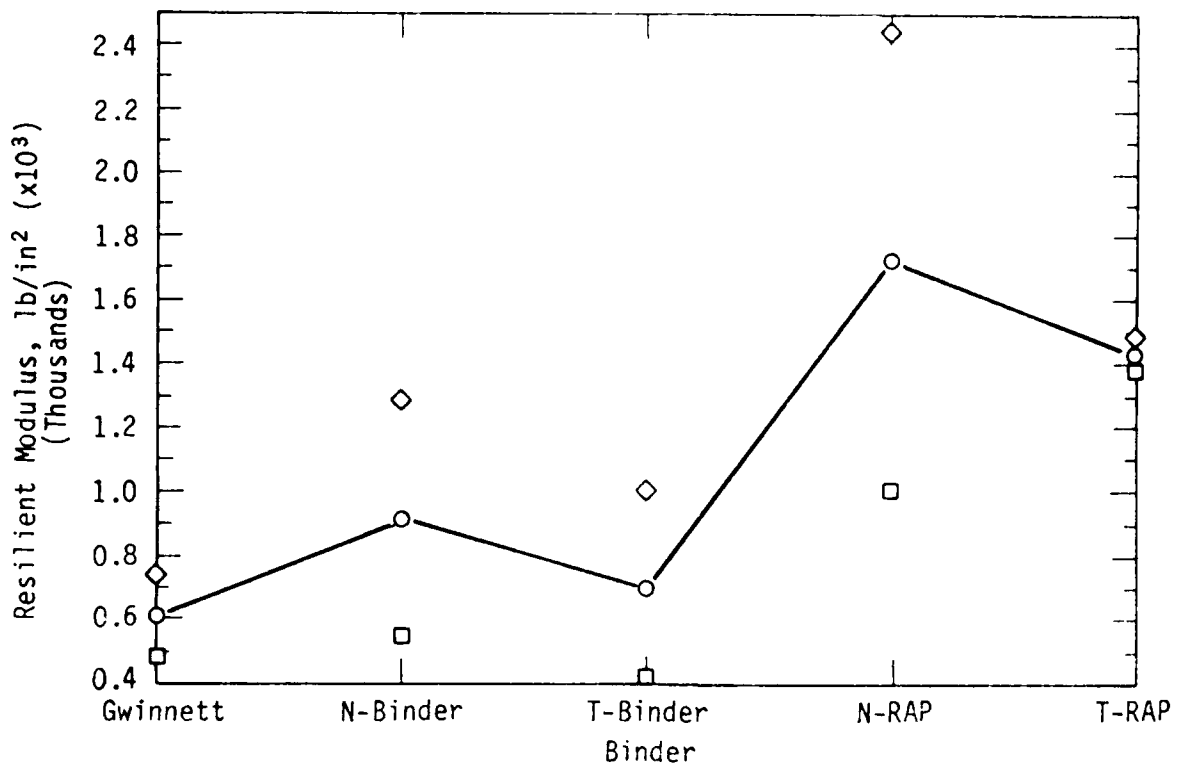(a) Dry Tensile Strength.

(b) Wet Tensile Strength.

Dry and Wet Tensile Strengths for Nellis (40/60) Formula Mixtures.

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(3) <u>Resilient Modulus</u>. Table 21 lists dry and wet resilient moduli data for NF mixtures. Figure 10 shows plots of mean dry and wet resilient moduli of NF mixtures. The recycled mixtures had comparable dry resilient moduli to that of the virgin mixture. The mixtures made with RAP binders had significantly higher dry resilient moduli than did the rest of the mixtures for the NF-aggregate system. Thus, the viscosity effect is further evident in these results.

Recycled systems were marginally stiffer than the virgin mixtures after water treatment. This marginal difference in stiffness was not statistically significant as shown by the Newman-Keuls results in Table 18. The NF Nellis RAP binder and NF Tyndall RAP binder mixtures were significantly stiffer than the recycled and virgin mixtures. The viscosity effect was still evident.

(4) <u>Resilient Modulus Ratio</u>. Mean resilient moduli ratios in percent were 59.7 for NF Gwinnett, 59.1 for NF Nellis blend, 76.1 for NF Tyndall blend, 60.8 for NF Nellis RAP binder, and 96.7 for NF Tyndall RAP binder. The ratio for NF Gwinnett virgin mixture is identical to the ratio for NF Nellis blend recycled mixture. However, the corresponding mean wet tensile strengths were 156.2 lb/in^2 for NF Gwinnett and 199.5 lb/in^2 for NF Nellis blend. For these mixtures, the wet resilient moduli were 364 k/in^2 for NF Gwinnett and 544 k/in^2 for NF Nellis blend. Thus, for the same viscosity binders and same aggregate, some mechanism must be responsible for leading to the observed significant differences in wet tensile strength, and/or wet resilient modulus.

In summary, recycled mixtures generally had slightly higher dry and wet resilient moduli. The resilient moduli ratios were about the same for the virgin and recycled systems and generally lower for the mixtures with RAP binders. The differences and/or similarities in the resilient moduli ratios are not matched by resilient moduli values. Thus, for binders with the same viscosity, the differences in mixture stiffness may be caused by some other mechanism. This mechanism is considered to be at the asphalt-aggregate interface.

(a) Dry Resilient Modulus.
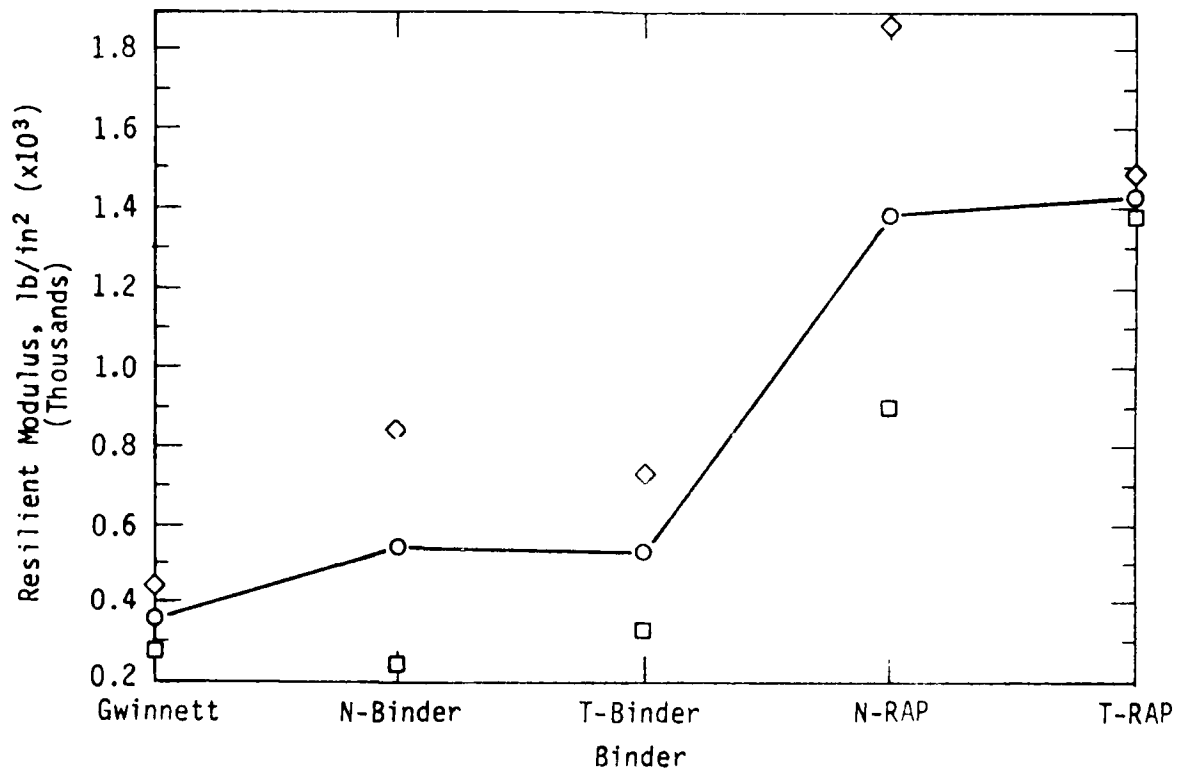
(b) Wet Resilient Modulus.

Figure 10. Dry and Wet Resilient Moduli for Nellis (40/60) Formula Mixtures.

The results suggest joint use of tensile strength ratio and wet tensile strength; resilient modulus ratio and wet resilient modulus should be considered in judging water susceptibility of bituminous mixtures.

d.   Miscellaneous Test Results

(1) Stripping observations. Observations of each wet mixture after the wet strength test were conducted to determine the level of stripping. The criteria used were defined in Section IV and employed a numerical ranking from 0 for no stripping, to 3 for severe stripping. Figures 11 and 12 show typical observations made in this study. This is a subjective rating with unestablished possibility for repeatability. The numerical ratings of stripping observations were listed in Tables 16 through 18.

(2) Duration for Vacuum Saturation. The duration for vacuum saturation was characteristic of each mixture. For instance, mixture GG Nellis RAP binder required 10 seconds, mixture RL Nellis RAP binder required 150 seconds, whereas mixture NF Nellis RAP binder required 840 seconds. The times in this example were the durations for each mixture to attain a level of moisture gain of about 65 percent of the specimen void volume. This level of moisture was precalculated for each mixture. The intent was to remain within the range given by NCHRP 274 method of 55 to 80 percent.

As a general observation, the duration for vacuum saturation increased from Grayson to Rome and to Nellis formula mixtures. Thus, it was easier to reach the precalculated saturation level for Grayson mixtures than for Rome and Nellis formula mixtures.

The durations are listed in Tables 19 through 21 containing mixture data.

e.   Model Analysis by Two-Way ANOVA

A generalized model of the form.

$$Y = L1 + L2 + (L1 \times L2)$$ (1)

Wet Specimens

Rate of Stripping

3   0   3

Nellis AFB   Rome Limestone   Grayson Granite Gneiss

Forty Percent RAP Aggregate

Sixty Percent Virgin Aggregate

Gwinnett AC-30

(a)   Gwinnett Virgin Binder.

Wet Specimens

Rate of Stripping

3   0   3

Nellis AFB   Rome Limestone   Grayson Granite Gneiss

Forty Percent RAP Aggregate

Sixty Percent Virgin Aggregate

Nellis Blend Binder
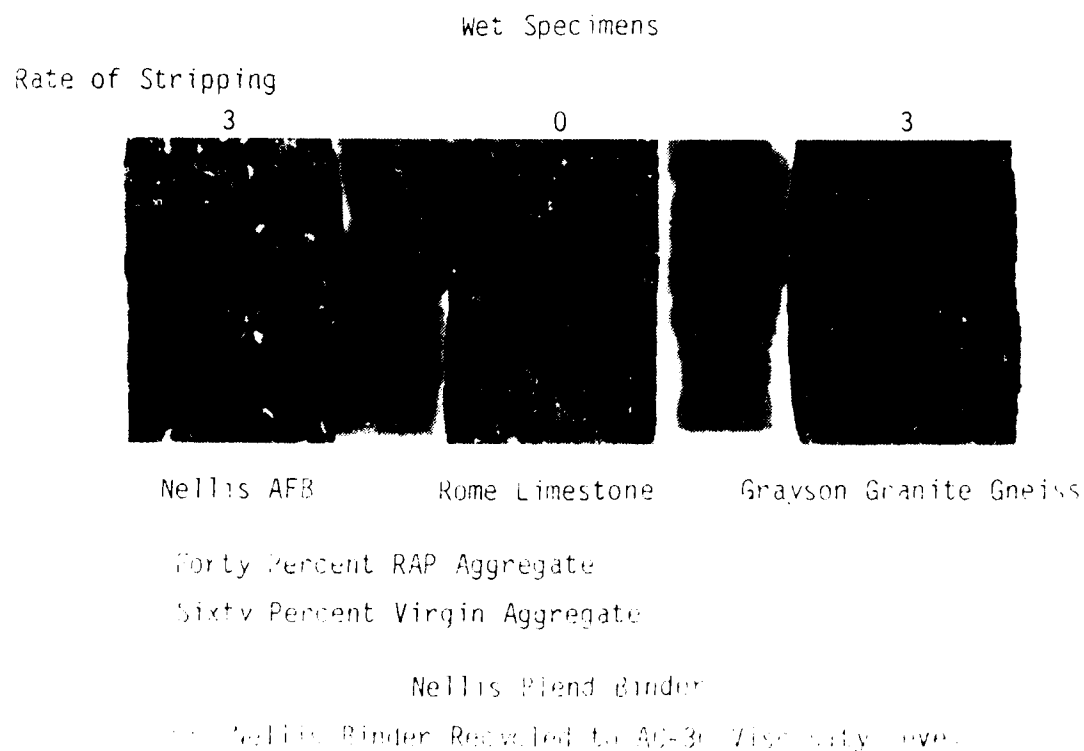
Nellis Binder Recycled to AC-30 Viscosity Level.
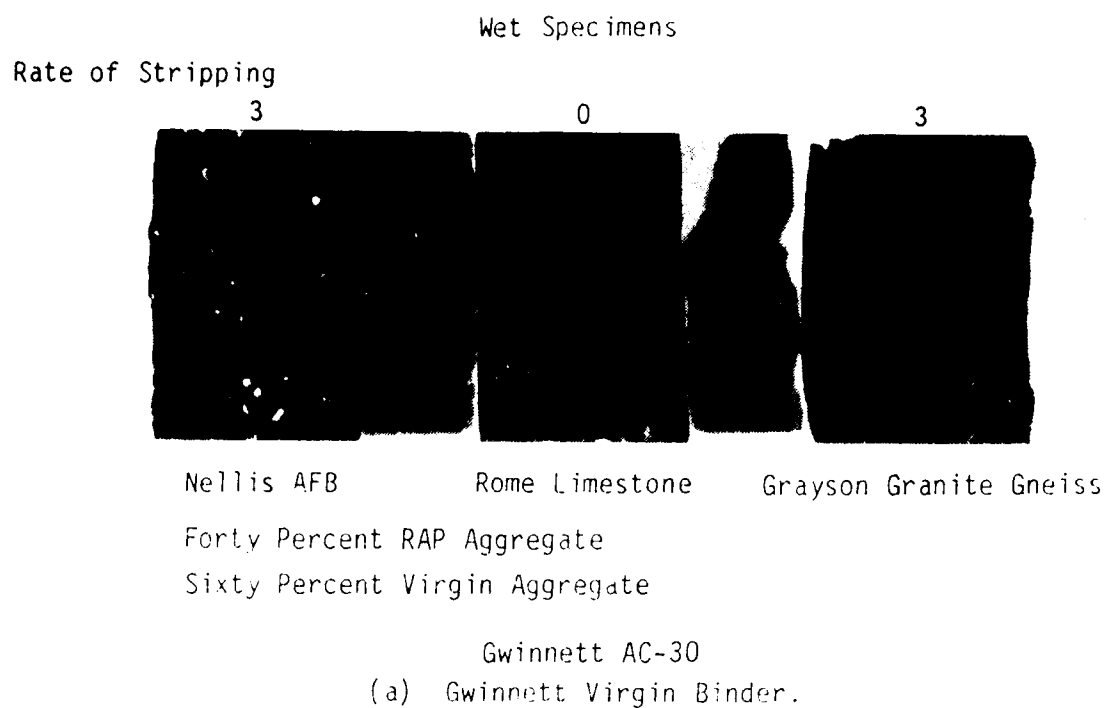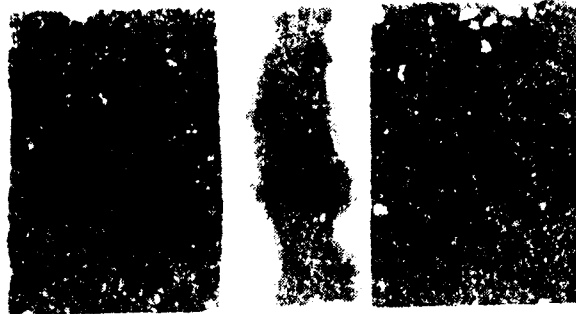
Figure 11.   Visual Stripping Comparison of Mixtures of Virgin and Recycled Binders with Various Aggregates.

Rate of Stripping

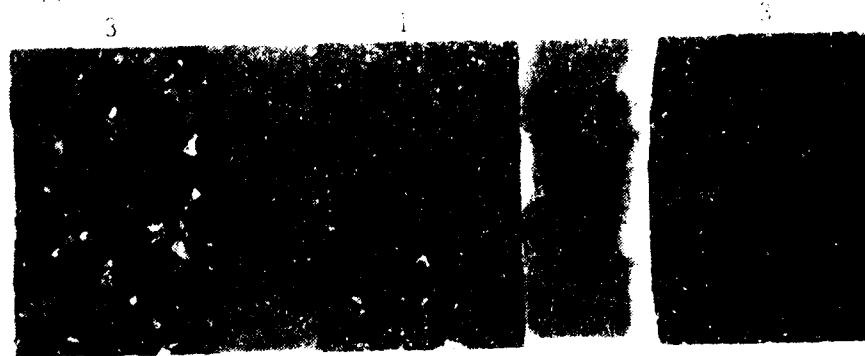Some Limestone   Grayson Granite Screen

Lydell and Binder

Mixture after 16 Cycles

Rate of Stripping

Nellis AFB   Some Limestone   Grayson Granite Screen

Forty Percent RAP Aggregate
Sixty Percent Virgin Aggregate

where

        Y = independent physical test
     L1 = aggregate type
     L2 = binder and
 L1xL2 = interaction term, and L1, L2 considered class variables

was employed in the two-way ANOVAs. The independent physical tests included in the analysis were dry tensile strength, wet tensile strength, dry resilient modulus, and wet resilient modulus.

Basic statistical theory of ANOVAs uses the F-distribution to determine statistical significance. If the value of F calculated is greater than a critical F value of the appropriate degrees of freedom, the term is deemed statistically significant. As seen in Table 22, all three components of the model are statistically significant to a high degree. The aggregate (L1), the binder (L2), and the interaction term (L1xL2) all have F values an order of magnitude greater than the critical F values. Therefore, the above model must be presented in its entirety without simplification by removing insignificant terms.

In summary, the results of the limited model analysis presented in Table 22 imply that characterization of either component of the model is important. The binder is most significant for dry and wet tensile strength. The interaction effects are most significant for dry and wet resilient modulii.

2. Microcalorimetry Test Results

The microcalorimetry data provide a measure of the strength of the bond formed initially between the asphalt and aggregate and the subsequent structuring of asphalt molecules near the aggregate surface. Upon addition of a compatible recycling agent to aged asphalt, highly agglomerated polar species in the binder are dispersed by the action of the recycling agent. This increased dispersion results in a greater number of free polar molecules that can bond to the aggregate surface. This effect should be manifested in an increase in the bonding energy of asphalt to aggregate. Each of the five binders was allowed to react with both the - #4 + #16 mesh

TABLE 22.   F VALUES DERIVED FROM THE TWO WAY ANOVA

Vari-able	Dry tensile strength	Wet tensile strength	Dry resilient modulus	Wet resilient modulus	F Critical 0.05%	F Critical 0.01%
N	45	45	45	45	na	na
L1	213	96	244	97	2.53	3.65
L2	318	550	280	143	2.37	3.34
L1xL2	254	463	292	177	1.75	2.20

Notes: N = Number of data points

na = Not applicable

L1 = Aggregate

L2 = Binder

L1xL2 = Aggregate-binder term

range and the - #16 + #50 mesh range of each aggregate. Inspection of the data for the - #4 + #16 mesh fraction revealed that a large amount of the measured heat released was due to friction of the larger aggregate against the microcalorimetry cell. Thus, this data will not be presented. Table 23 summarizes the microcalorimetry data for the - #16 + #50 mesh fractions of the aggregate with the binders studied.

In Figure 13, the heat released is plotted for each of the three aggregate systems studied. There is a clear ordering of Nellis > Rome > Grayson. This ordering does not follow the trend in surface areas for the - #16 + #50 fractions. This effect appears to be due to variations in the samples for which the surface areas were measured. The porosity measurements of the - #16 + #50 fractions showed the Nellis material had the only porosity high enough to measure and had close up significant pore volume at pore radii less than 1000 Å. The Nellis - #200 fraction had the highest surface area due to the large pore volume at pore radii less than 1000 Å. Thus, the surface area measurement of the Nellis - #16 + #50 fraction was not representative of the entire Nellis material.

Figure 14 shows the relationship between mean dry tensile strength and and the surface area of an entire Marshall briquette. The surface area of the briquette was calculated from the measured surface area and the mass of a particular sieve range used in constructing a briquette. The surface area contribution from material greater than sieve #4 diameter was considered negligible. A Newman-Keuls analysis was conducted at the 95 percent confidence level to search for a ranking of the mean dry tensile strengths for each binder system (Table 24). The Newman-Keuls analysis of the Gwinnett asphalt mixes shows a tensile strength ranking of Nellis > Rome > Grayson. For the Tyndall blend binder, the Newman-Keuls analysis finds no significant difference in the dry tensile strength between the Grayson and Rome aggregate. However, the Nellis material has a significantly higher mean dry tensile strength than the other aggregates. For the Nellis blend binder, the Newman-Keuls analysis provides a clear ranking of Nellis > Rome > Grayson for the mean dry tensile strengths of the briquettes. Thus, there is a definite relationship between the surface area of an aggregate and the mean dry tensile strength of a mixture containing that aggregate.

TABLE 23. BONDING ENERGY MEASUREMENTS FOR - #16 + #50 AGGREGATE
           FRACTIONS WITH EACH BINDER

Binder	Grayson		Rome		Nellis 40/60	
	mcal/ g-2 hr	tail hgt	mcal/ g-2 hr	tail hgt	mcal/ g-2 hr	tail hgt
G	82	0.50	167	0.66	224	0.95
NR	44	0.28	100	0.40	189	0.85
TR	46	0.20	109	0.40	195	0.96
NB	78	0.12	174	0.13	280	0.39
TB	68	0.13	177	0.41	316	0.95

NOTES: G = Gwinnett AC-30

         NB = Nellis blend

         TB = Tyndall blend

         NR = Nellis RAP

         TR = Tyndall RAP

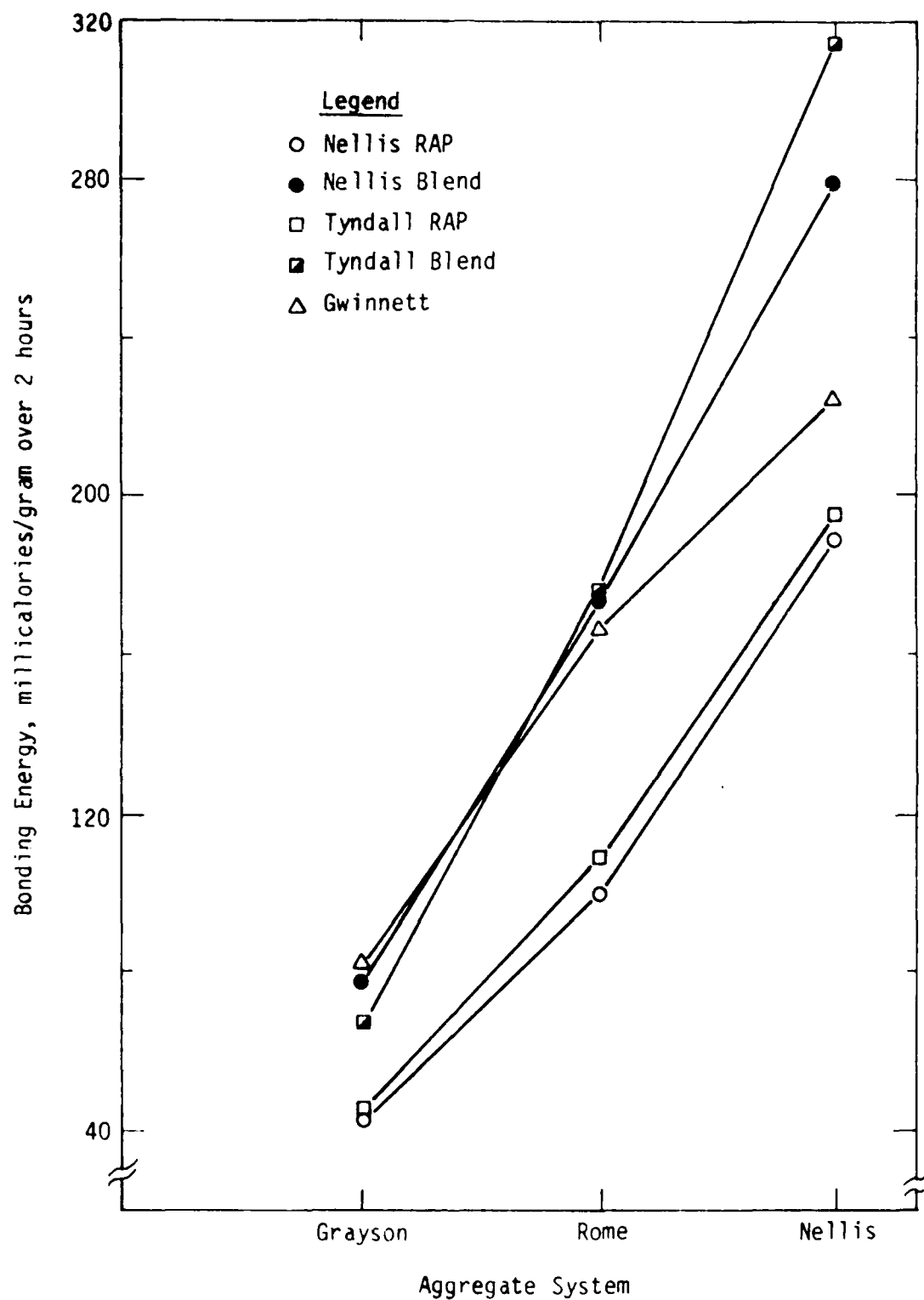   hgt = height

          g = gram

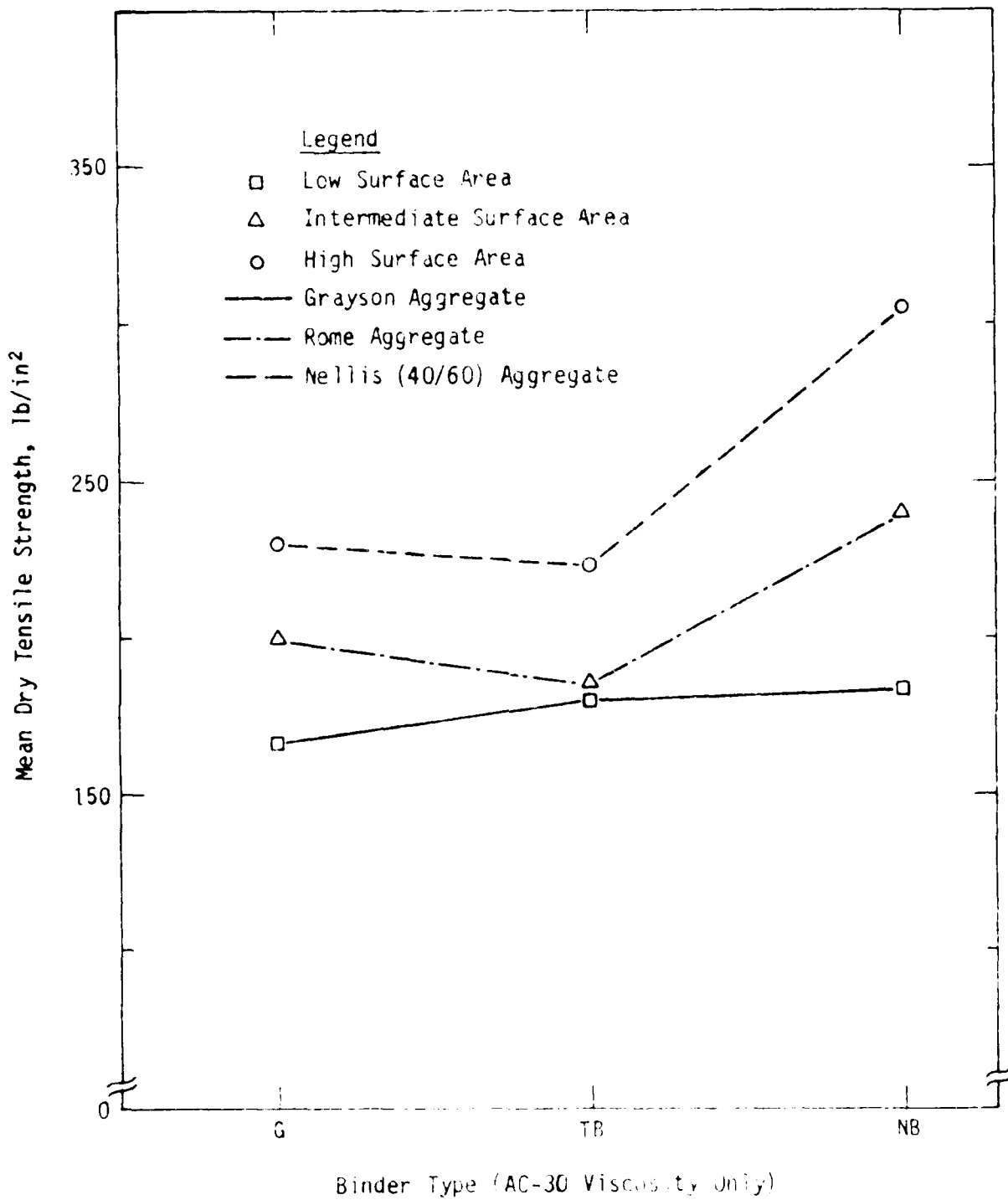Figure 13. Total Heat Release of each Binder for the Three Aggregate Systems.

Figure 14. Comparing Mean Dry Tensile Strength to the Surface Area of Aggregates in a Marshall Specimen Using Same Viscosity Binders.

TABLE 24. NEWMAN-KEULS RANKING FOR DRY TENSILE STRENGTHS OF BINDERS WITH VISCOSITIES AT[a] AC-30

Aggregate	Gwinnett		Tyndall		Nellis	
	Mean	Rank	Mean	Rank	Mean	Rank
Nellis	229.63	A	223.37	A	305.67	A
Rome	198.93	B	185.73	B	238.67	B
Grayson	167.00	C	179.63	B	183.33	C

[a]All ranking was determined by the Newman Keuls algorithm for determining significant differences between means.

The data presented in Figure 14 show an increase in heat release for the recycled systems over those of the RAP binders and recycling agents. This result is in accordance with the prediction that when the asphaltenes are dispersed, the released molecules are freed to compete with the polars for bonding sites at the aggregate interface. As the number of bonding sites between molecules and the increased competition occurs... In Figure 15, the relationship between the heat release and the percentages of selected Clay-Gel fractions... the recycling agent lowers the asphaltene content by either dispersing the asphaltenes into "molecules" with a corresponding increase in the polar fraction. As this occurs, the bonding energy of asphalt to aggregate increases. This increase in released heat is also manifested in thermodynamic state of peptization, as shown in Figure 16. In each case, an increase in the bonding energy follows an increase in the state of peptization of the binder.

The sum of the asphaltene and the polars demonstrates the dominance of the asphaltene fraction in determining the bonding energy increase (Figure 15). Consider several asphaltene molecules that can bond to an aggregate surface and assume each of the asphaltenes to be a simple collection of polar molecules. Upon addition of a recycling agent, assume only one polar molecule is freed from the asphaltenes and bonds to the aggregate surface. If the remaining asphaltene molecules are still able to bond to the surface then an increase in the bonding energy will depend more strongly on the asphaltene fraction, not the polars.

In summary, the beneficial effects of the recycling agent on the solution properties of the RAP binders are manifested by an increase in bonding energy of the recycled binders. In most cases, the heat released for recycled systems is greater than that for the corresponding virgin system. The bonding energy is dependent on the surface area, type, chemical composition of the aggregate, and binder properties.

D.   RESULTS SUMMARY

The Nellis and Rome aggregates ... are classed as basic aggregates ... is classed as acidic. The ... Rome aggregate interface ...
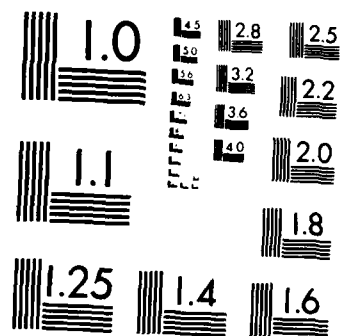
MICROCOPY RESOLUTION TEST CHART
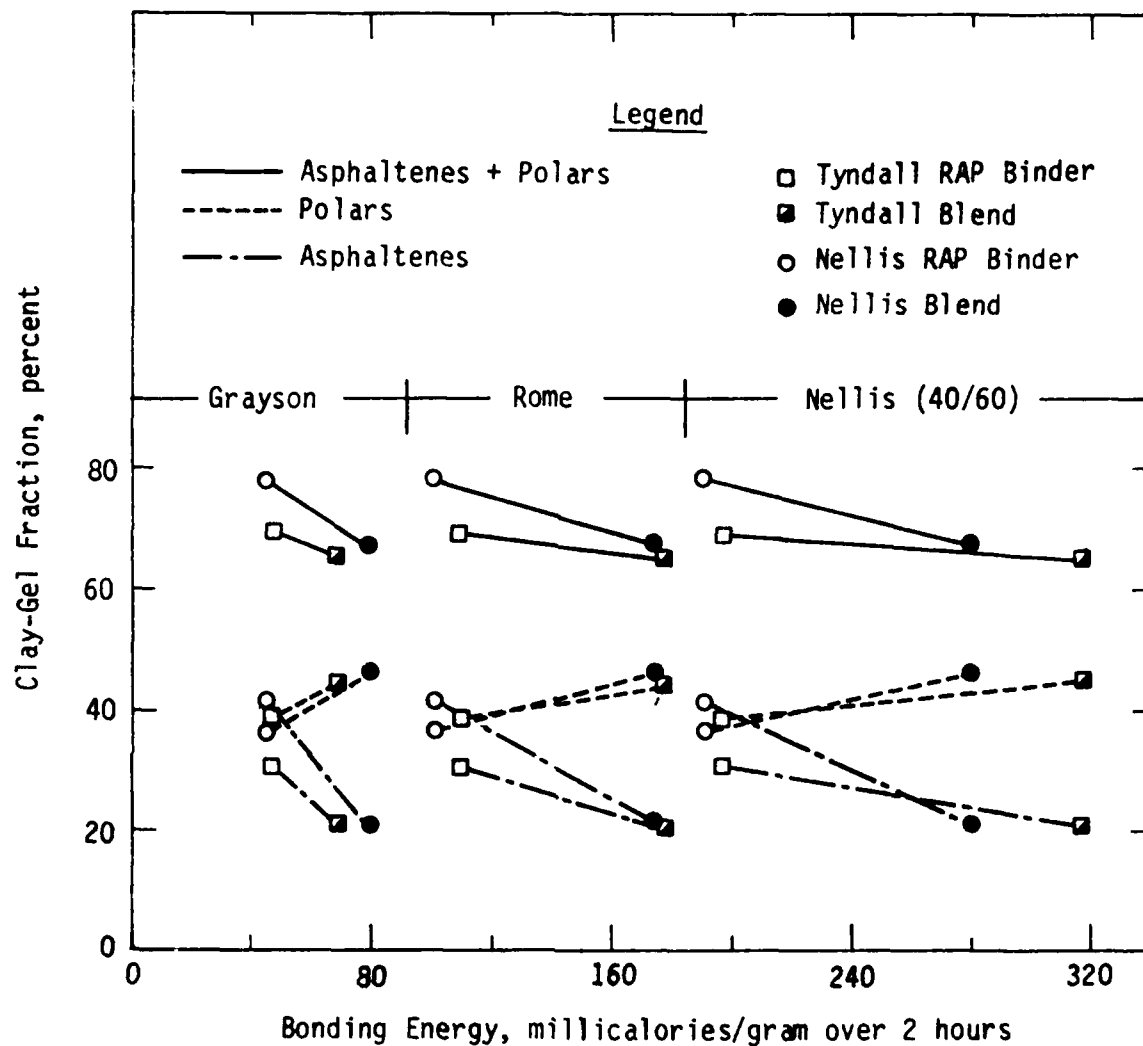
NATIONAL BUREAU OF STANDARDS 1963 A

Figure 15. Relationship of Microcalorimetry Heat Release with the Change in Clay-Gel Fractions Upon Recycling.
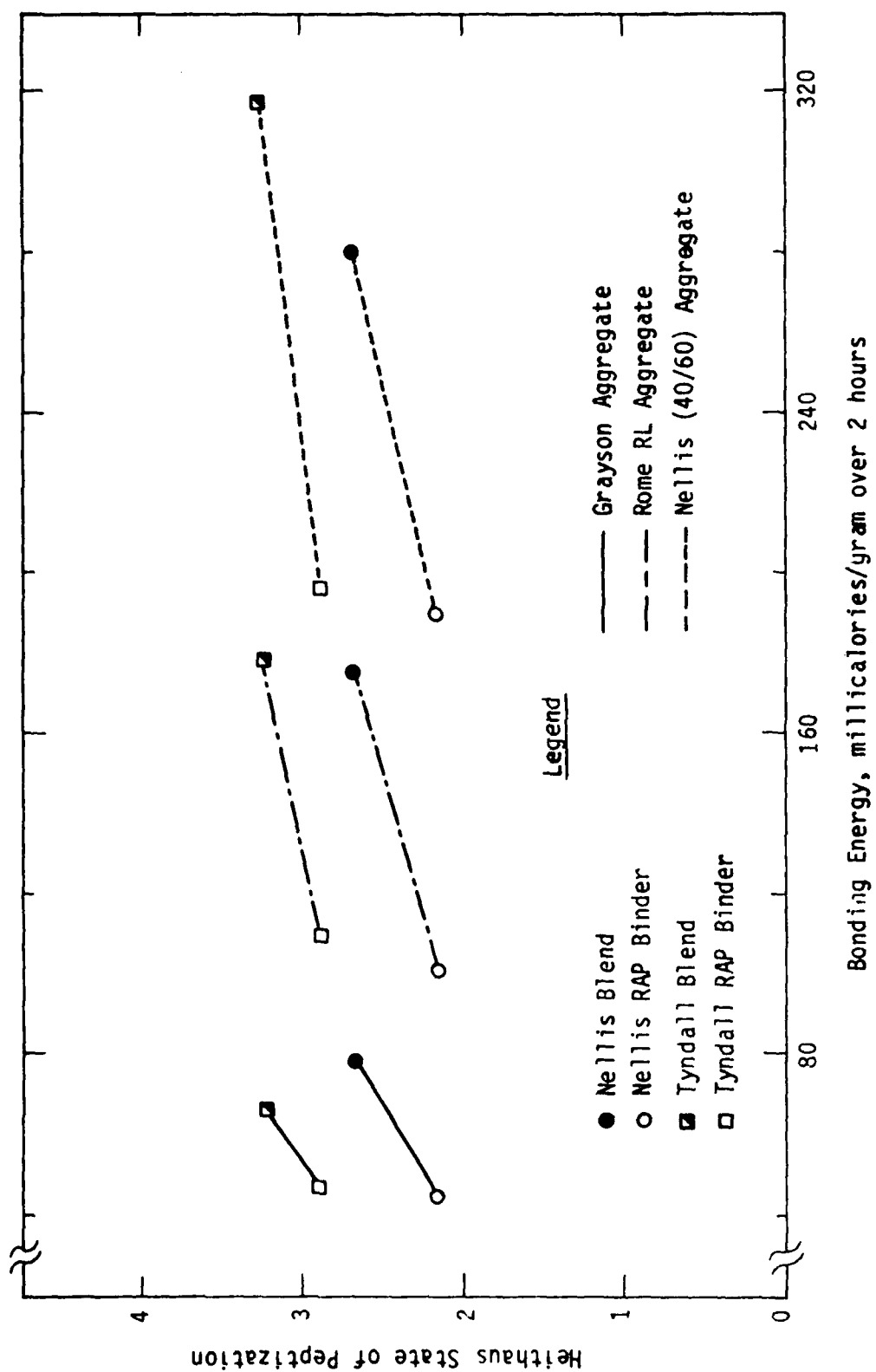
Figure 16. Relationship of Microcalorimetry Heat Release to the Change in the Heithaus State of Peptization upon Recycling

The addition of a recycling agent restores the RAP binder to its original consistency with physical properties similar to that of the Gwinnett virgin binder. The action of the recycling agent raises the state of peptization of the RAP binder but not necessarily to the magnitude of the virgin binder. The asphaltene peptizability and maltene peptizing power also increase with addition of the recycling agent. These changes in the RAP binder are manifested as decreases in the asphaltenes and increases in the polar fractions as measured by the Clay-Gel technique.

The bonding energies of the Nellis aggregate system are the highest of the three aggregate systems, probably due to the higher surface area of the Nellis material. Upon addition of the recycling agent, the state of peptization of the binder increases with a resulting increase in the bonding energy of binder to aggregate. This bond energy increase is also related to the changes in the Clay-Gel fractions that occur with addition of a recycling agent.

The three mixture systems were prepared using the same binder content, gradation, mixing and compaction temperatures. Thus, the following general summaries are made from the results presented and discussed above.

- Recycled mixtures using the three aggregates had statistically higher dry tensile strength than corresponding mixtures made with the virgin binder. Overall, the strength and stiffness of the recycled mixtures were lower than those of the RAP binder mixtures due to the viscosity effect. The dry tensile strength of the recycled mixtures is observed to be related to the surface area of the aggregate.

- Recycled mixtures generally had significantly higher wet tensile strengths than control mixtures made with the virgin binder. The viscosity efffect could not explain the results in these two observations. Thus, some other mechanism, most probably the asphalt-aggregate interaction, may be the cause.

- Tensile strength ratio values for recycled mixtures were predominantly higher than those for control mixtures made with the virgin binder.

- The dry tensile strengths of mixtures made with RAP binders were, in general, significantly higher than strengths of corresponding recycled and virgin binder mixtures. The viscosity effect was evident.

- The wet tensile strengths of RAP binder mixtures varied from comparable to statistically higher than recycled mixtures. Thus, the viscosity effect was not as pronounced as it was with the dry systems.

- The dry resilient moduli of recycled mixtures were marginally higher than similar values for virgin binder mixtures.

- The wet resilient moduli of recycled mixtures were, in general, significantly higher than similar values of mixtures made with virgin binder. The viscosity factor could not explain this result.

- Overall, recycled mixtures sustained the action of water better than the virgin and RAP binder mixtures. This observation is based on results of marked improvements in wet tensile strength, wet resilient moduli, and corresponding tensile strength ratio and resilient modulus ratio values compared to similar measurements on mixtures with the virgin binder used in this study.

Results of strip rating of mixtures were discussed with 0 indicating nonstripped and 3 severely stripped mixtures. The duration of presaturation time was reported to be characteristic of the mixture. Recycled mixtures showed longer durations of presaturation than virgin binder mixtures.

Finally, a model was presented from which independent analysis of aggregate and or binder was found to be inseparable. The three components of the model, namely aggregate, binder, and binder-aggregate term were all significant.

SECTION VI
CONCLUSIONS

A. AGGREGATES

1. Aggregates used in this study were within range of acceptable specific gravities for paving operations and were nonabsorptive for paving requirements.

2. The Rome and Nellis carbonaceous limestones withstood water damage better than the siliceous Grayson granite.

3. Aggregates with higher surface areas resulted in mixes with higher dry tensile strength.

4. Aggregates with higher surface areas (Rome and Nellis) had higher bonding energies than the aggregate with the least surface area (Grayson).

5. The Nellis aggregate had the lowest concentration of water-soluble ions per area, the Rome material had the intermediate value, and Grayson the highest. The susceptibility to water damage increased with the concentration of water-soluble ions per area.

B. BINDERS

1. Addition of the recycling agent to the RAP binders resulted in lowering the asphaltene content, raising the amount of polars and the state of peptization of the resulting blend. An increase in the bonding energy of the binder to the aggregate followed each of the above trends.

2. A modifier compatible to two RAP binders from different climatic environments was determined using the modifier selection criteria established in Reference 12 and as modified in Reference 47.

C. MIXTURES

1. The difference in dry and wet tensile strengths between virgin and recycled mixtures was statistically significant in most cases. The same was true for the resilient modulus values.

2. A hardening effect was observed in RAP binders, as dry tensile strengths and dry resilient moduli of mixtures made with RAP binders were significantly higher than those of recycled and virgin mixtures.

3. Tensile strength ratio values for mixtures made with the Grayson aggregate and recycled binders were significantly higher than those of virgin mixtures. This result was also true for resilient modulus ratios.

4. Tensile strength ratio values for mixtures made with Rome and Nellis aggregates and recycled binders were comparable to tensile strength ratio values for mixtures with virgin binder. This result was also true with resilient modulus ratios.

5. The results suggest that the use of tensile strength ratio and or resilient modulus ratio should be supplemented by tensile strength and or resilient modulus in wet conditions for adjudging water susceptibility of bituminous mixtures.

6. Recycled mixtures were significantly less susceptible to the action of water than mixtures made with virgin and RAP binders.

7. Recycled mixtures had higher bonding energies than virgin mixtures suggesting that the recycled systems may show higher strength than virgin mixes. This is generally supported by results of this study.

SECTION VII
RECOMMENDATIONS

1. Efforts should be made to develop and verify criteria using tensile strength ratio or resilient modulus ratio and tensile strength or resilient modulus for discerning moisture susceptibility of a bituminous mixture.

2. The results of this study should be expanded by examining a variety of other stripping aggregates to strengthen the observations made. The results of such an investigation may be used to develop procedures such as psychrometry for routine evaluation of aggregates.

SECTION VIII
RECYCLING GUIDELINES

A. BASIC MATERIALS

1. Aggregates

Aggregates (virgin and RAP) should have properties meeting local or project specifications. In addition, aggregates may be evaluated for:

- Basic and or acid properties
- Water-soluble ions
- Surface area on fine fractions (- #16 + #50)

2. Binders

a. RAP Binders

These materials are in the pavement and are generally of unknown origin. A representative sample of RAP materials should be extracted and recovered using standard procedures. The procedures currently used are:

- ASTM D 2172-81
- ASTM D 1856-84
- Modifications such as use of a Rotovap system. This modification was used in this study.

The recovered binders should be tested for:

- Viscosity at two temperatures (minimum)
- Penetration at 77 °F and 39.2 °F.
- Composition properties using modified Clay-Gel method (AFESC-1)
- Solubility properties using modified Heithaus method (AFESC-2)

b.　Modifiers

A modifier should be selected using the criteria developed and referenced in this report. The criteria list:

- Physical property requirements
- Composition property requirements
- Solubility property requirements

c.　Blends

Blends should be made and tested as described for RAP binders. In addition, blends should be tested for age-hardening using an RTFO. The aged residue should be retested using the same test procedures. The test results should be checked for the following using the RAP binder as the reference material:

- Blend aging index (about 3.0 or less preferred)
- Net reduction in RAP asphaltene content
- Net increase in polar compounds
- Net increase in state of peptization, and other solubility properties
- Blends should meet local binder specifications

d.　Virgin Binders

Virgin binders are often used to meet additional film requirements due to the use of new aggregate in the mix. These binders should meet the requirements in the local or project specifications. In addition, they should be analyzed for composition and solubility properties using the procedures discussed above.

B.　MIXTURES

1.　Performance of recycled mixtures

a.　Tensile strength and tensile strength ratio

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The results from this study suggest that using tensile strength ratio as the sole criteria for judging water susceptibility of bituminous mixtures may be misleading. The argument is based on observations in this study showing that mixtures with a high tensile strength ratio value had lower tensile strength compared to mixtures with lower tensile strength ratio and higher tensile strength. In terms of susceptibility to tensile deformations, the mix with a higher tensile strength and lower tensile strength ratio may offer better resistance than a mix with lower tensile strength and higher tensile strength ratio.

Results from recent unpublished field investigations have shown that field pavement sections with a tensile strength ratio both greater than and lower than 0.7 are slightly stripped. This dual standard about the 0.7 ratio suggests that more mixture properties may need to be identified and included in the selection process of water-susceptible mixtures.

Thus, the results of this research suggest that in addition to the use of tensile strength ratio, tensile strengths should be compared between mixtures when choosing a water-resistant mixture. Recycled mixtures should be compared with conventional virgin mixtures or with mixtures made with alternate modifying products.

b.   Resilient modulus and resilient modulus ratio

Resilient moduli values which are generally considered for pavement applications range from 100 to 1,500 k/in^2. Thus, results of this research suggest that wet resilient moduli of recycled mixtures should be accepted if they range from 100 to 1,000 k/in^2. This suggested range may be used in addition with the resilient modulus ratio.

The argument for using a combined information base of a ratio and a stiffness remains the same as presented for tensile strength above.

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APPENDIX A

METHOD OF TEST FOR DETERMINING THE EFFECT OF
MOISTURE AND ANTISTRIPPING ADDITIVES ON ASPHALT
CONCRETE PAVING MIXTURES

This appendix consists of text taken from a portion of Reference 39 and is reproduced as printed.

METHOD OF TEST FOR DETERMINING THE EFFECT OF MOISTURE AND ANTISTRIPPING ADDITIVES ON ASPHALT CONCRETE PAVING MIXTURES

1. Scope

This method contains procedures for preparing and testing specimens of asphaltic concrete for purposes of measuring the effect of water, or the effectiveness of antistripping additives on the tensile strength of the paving mixture. The method is applicable to dense mixtures such as those appearing in the upper half of Table 3, ASTM Specification D 3515. The method can evaluate the effect of moisture with or without additives, the effect of liquid antistripping additives which are added to the asphalt cement, or pulverulent solids such as hydrated lime or portland cement which are added to the mineral aggregate.

2. Applicable Documents

2.1. ASTM Standards

- D 979 Method for Sampling Bituminous Paving Mixtures
- D 1559 Test for Resistance to Plastic Flow of Bituminous Mixtures by Marshall Apparatus
- D 2041 Test for Theoretical Maximum Specific Gravity of Bituminous Paving Mixtures
- D 2726 Test for Bulk Specific Gravity of Compacted Bituminous Mixtures Using Saturated Surface-Dry Specimens
- D 3203 Test for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures
- D 3515 Specification for Hot-Mixed, Hot-Laid Bituminous Paving Mixtures
- D 3549 Test for Thickness or Height of Compacted Bituminous Paving Mixture Specimens
- D 3665 Practice for Random Sampling of Construction Materials
- D 4123 Method of Indirect Tensile Test for Resilient Modulus of Bituminous Mixtures

3. Significance and Use

This method can be used to test asphaltic concrete mixtures in conjunction with mixture design testing to determine whether or not moisture damage is severe enough so that an additive should be considered, and if it is severe enough, to determine whether or not an antistripping additive is effective and what dose of additive is most effective. It can also be used to test mixtures produced at plants to determine the severity of moisture damage and the effectiveness of additives under conditions imposed by construction in the field. Finally, it can be used to test cores from completed pavements of any age to determine the severity of moisture damage and the effectiveness of additives under conditions of exposure and service in the field.

4. Summary of Method

4.1. To determine the severity of moisture damage and decide whether or not an additive should be considered, a set of laboratory-compacted specimens conforming to the job-mix formula without additive is prepared. The specimens are compacted to a void content corresponding to void levels expected in the field, usually in the 6 to 8 percent range. The set is divided into two subsets of approximately equal void content, and one subset is maintained dry, while the other subset is saturated with water and moisture conditioned. The tensile strength of each subset is determined by the tensile splitting test. The severity of moisture damage is indicated by the ratio of the tensile strength of the wet subset to that of the dry subset.

4.2. To determine the effectiveness of an antistripping additive a set of specimens containing additive but otherwise the same as the set in Section 4.1 is prepared and tested, and the severity of the moisture damage is determined in the manner described in Section 4.1. The effectiveness of the additive is indicated by the improvement in the wet-to-dry ratio of the set containing additive compared to the set without additive. The effect of

additive dosage may be estimated by repeating the set with different additive dosages.

4.3. To determine the severity of moisture damage or the effectiveness of an additive in mixture produced in an asphalt plant in the field, specimens are laboratory compacted to field level void content, divided into wet and dry subsets, and the severity of moisture damage or the effectiveness of the additive is determined as in Section 4.2.

4.4 To determine the severity of moisture damage or the effectiveness of an additive in specimens cored from a pavement, cores are maintained at in-place moisture content until tensile strength is measured. This strength may be compared to the tensile strength determined previously before moisture damage occurred.

5. Apparatus

5.1. Equipment for preparing and compacting specimens from Method D 4123.

5.2. Vacuum pump or water aspirator, manometer or vacuum gauge, and container, preferably Type D, from Method D 2041

5.3. Balance and water bath from Method D 2726

5.4. Water bath or oven capable of maintaining a temperature of 140 F for 24 hours.

5.5. Loading jack and ring dynamometer from Method D 1559, or a mechanical or hydraulic testing machine capable of maintaining the required strain rate and measuring load with suitable precision.

5.6. Loading strips from Method D 4123

6. Preparation of Laboratory Test Specimens

6.1. At least six specimens shall be made for each test, three to be tested dry and three to be tested after saturation and moisture conditioning.

6.2. Specimens 4 in. in diameter and 2.5 in. thick are usually used. Specimens of other dimensions may be used if desired and should be used if aggregate larger than 1 in. is present.

6.3. When 4-in. × 2.5-in. specimens are used, mixtures shall be prepared in batches large enough to make at least 3 specimens. When larger specimens are used, batches may be prepared for each specimen. If theoretical maximum specific gravity is to be determined, the batch should be large enough to provide the specimen for that purpose also.

6.4. When a liquid antistripping additive is used, the asphalt cement in sufficient quantity for one batch shall be heated to 300 F in a closed one quart can in an oven. The required quantity of additive shall be added. Immediately lower a mechanical stirrer to within 1 in. of the bottom of the container, and mix the contents for 2 min. Maintain the treated asphalt cement at 300 F in the closed can until it is used. If the treated asphalt cement is not used on the same day in which it is prepared, or if it is allowed to cool so that it would require reheating, it shall be discarded.

6.5. When a pulverulent solid antistripping additive is used, the batch of mineral aggregate shall be dried, composited, and heated to 300 F The required quantity of additive shall be added to the aggregate, and the entire mass shall be thoroughly mixed until a uniform distribution of additive has been achieved. Care

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...be taken to minimize loss of additive to the atmosphere in the form of dust. After mixing, maintain the treated aggregate at the temperature required for mixing until it is used.

6.6. Prepare, mix, and compact specimens in accordance with Method D 4123 and Sections 6.6.1 and 6.6.2.

6.6.1. After mixing, stabilize mixture temperature at the required compaction temperature in a closed container in an oven for from 1 to 2 hours.

6.6.2. Compact specimens to 7 ± 1 percent air voids, or a void level expected in the field. This level of voids can be obtained by adjusting the static load in double plunger compaction; the number of blows in Marshall hammer compaction; the foot pressure, number of tamps, leveling load, or some combination in kneading compaction; or the number of revolutions in gyratory compaction. The exact procedure must be determined by trial for each mixture.

6.6.3. Cool specimens to room temperature as rapidly as possible in a stream of moving air, extract from molds, and proceed with Section 9 immediately if possible, but within 24 hours at most.

7. Preparation of Field Specimens

7.1. Select a truck to be sampled in accordance with Practice D 3665.

7.2. Secure a sample from the truck at the plant in accordance with Method D 979.

7.3. Stabilize mixture temperature to approximately the temperature found in the field when rolling begins. Maintain this temperature in a closed container, in an oven if necessary, for approximately the time lapse between mixing and the start of actual rolling.

7.4. Compact specimens in accordance with Section 6.6.2, and cool and extract from molds in accordance with Section 6.6.3.

7.5. If specimens are not to be compacted in the field laboratory, place the samples in a sealed container, transport to the laboratory, and reheat to the temperature required in Section 7.3. Then proceed with Section 7.4.

8. Preparation of Core Test Specimens.

8.1. Select locations to be sampled on the completed pavement or pavement layer in accordance with Practice D 3665.

8.2. Core at the selected locations in accordance with Method D 979. A wet coring process should be used, and the periphery of the core should be blotted dry immediately after it is taken. Wrap the core in plastic wrap or otherwise protect it to maintain field moisture content until the test layer of the core is separated.

8.3. Separate core layers as necessary by sawing or other suitable means. A wet sawing process is preferred, and the periphery of the test layer of the core should be blotted dry immediately after it is sawn. Wrap the test layer in plastic wrap or otherwise protect it to maintain field moisture content until it is tested.

9. Procedure

9.1. Determine the theoretical maximum specific gravity by Method D 2041.

9.2. Determine specimen thickness by Method D 3549.

9.3. Determine the bulk specific gravity by Method D 2726, and express the volume of the specimen in cubic centimeters. The term (B-C) in Method D 2726 is the volume of the specimen in cubic centimeters.

9.4. Calculate air voids by Method 3203, and express the volume of air in cubic centimeters. The volume of air is the volume of the specimen from Section 9.3 multiplied by the percentage air voids.

9.5. Sort specimens into two subsets so that average air voids of the two subsets are approximately equal. Store the subset to be tested dry at room temperature.

9.6. Saturate the subset to be moisture conditioned with distilled water at room temperature. If it is difficult to reach the minimum degree of saturation of 55 percent required in Section 9.6.3, the water used to saturate may be heated up to 140 F.

9.6.1. Saturate by applying a partial vacuum such as 20 in. Hg for a short time such as 5 min.

Note 1: Experiments with partial vacuum at room temperature indicate that degree of saturation is very sensitive to the magnitude of the vacuum and practically independent of the duration. The level of vacuum needed appears to be different for different mixtures.

9.6.2. Determine bulk specific gravity by Method D 2726. Determine the volume of absorbed water by subtracting the air dry weight of the specimen found in Section 9.3 from the saturated surface dry weight of the saturated specimen found in Section 9.6.2.

9.6.3. Determine the degree of saturation by dividing the volume of absorbed water found in Section 9.6.2 by the volume of air voids found in Section 9.4 and expressing the result as a percentage. If the volume of water is between 55 and 80 percent of the volume of air, proceed to Section 9.7. If the volume of water is less than 55 percent, repeat the procedure beginning with Section 9.6.1 using a slightly higher partial vacuum. If the volume of water is more than 80 percent, the specimen has been damaged and is discarded.

Note 2: If the average air voids of the saturated subset is less than 6.5 percent, saturation of at least 70 percent is recommended.

9.7. Moisture-condition the saturated specimens by soaking in distilled water at 140 F for 24 hours.

9.8. Adjust the temperature of the moisture-conditioned subset by soaking in a water bath for 1 hour at 77 F.

9.9. On moisture-conditioned subset, measure thickness by Method D 3549, and determine bulk specific gravity by Method D 2726.

9.9.1. Determine water absorption and degree of saturation in accordance with Section 9.6.2 and Section 9.6.3. Saturation exceeding 80 percent is acceptable in this step.

9.9.2. Determine swell of saturated specimens by dividing the change in specimen volumes found in Sections 9.6.2 and 9.3 by the specimen volume found in Section 9.3. Determine swell of conditioned specimens by dividing the change in specimen volumes found in Sections 9.9 and 9.3 by the specimen volume found in Section 9.3.

9.10. Adjust temperature of dry subset by soaking in a water bath for 20 min at 77 F.

9.11. Determine tensile strength at 77 F of both subsets

9.11.1. Apply diametral load in accordance with Method D 4123 at 2.0 in. per minute until the maximum load is reached, and record the maximum load.

9.11.2. Continue loading until specimen fractures. Break open and estimate and record stripping, if any

9.11.3. Inspect all surfaces, including the failed faces, for evidence of cracked or broken aggregate, and record observations

10. Calculations

10.1. Tensile Strength

$$S = 2P / \pi t D$$

where

S = tensile strength, psi.
P = maximum load. lb
t = specimen thickness immediately before tensile test, in , and
D = specimen diameter. in

10.2. Tensile Strength Ratio

$$TSR = (S_m / S_d)100$$

where

TSR = tensile strength ratio, percent.
S_m = average tensile strength of moisture-conditioned subset, psi, and
S_d = average tensile strength of dry subset, psi.

11. Report

11.1. Average room temperature at which any measurements are made

11.2. Number of specimens in each subset

11.3. Average degree of saturation after saturating and after moisture conditioning

11.4. Average swell after saturating and after moisture conditioning

11.5. Tensile strength of each specimen in each subset

11.6. Tensile strength ratio

11.7. Results of estimated stripping observed when specimen fractures.

11.8. Results of observations of fractured or crushed aggregate

12. Precision

12.1. Precision of the method is under study

12.2. Tests on one moisture-conditioned moisture containing additive in one laboratory indicate that the difference in tensile strength between duplicate specimens should not exceed 25 2 psi.

END

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MARCH, 1988

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